

Operation Manual

Lyovapor™ L-250 Basic / L-250 Pro



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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.
- \Rightarrow 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.
orginar	

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 Intended use

The instrument is used for freeze-drying solid materials in ampoules, vials, dishes, round-neck or wide-neck flasks and trays and is exclusively intended for that purpose. The instrument can be used in laboratories for the following tasks:

- Sublimating and re-sublimating water-based samples
- Sublimating and re-sublimating samples containing organic solvents, acids and bases

2.2 Use other than intended

Use of any other kind than that described in Chapter 2.1 "Intended use", page 8 and any application that does not comply with the technical specifications (see Chapter 3.5 "Technical data", page 19) constitutes use other than intended. In particular, the following applications are not permissible:

- Use of the instrument in an environment with a potential risk of explosion or areas which require explosion-safe apparatus.
- Use of the instrument for processing substances outside of research and development.
- Production and processing of substances that can lead to spontaneous reactions, such as explosives, metal hydrides or solvents that can form peroxides.
- Processing samples not under a vacuum.
- Processing too many samples and overloading the instrument, see Chapter 3.5 "Technical data", page 19.
- Processing with explosive gas mixtures.
- Drying samples with high solvent concentrations without special safety precautions.
- Use of acids and alkalis without first checking material compatibility.
- Use of solvents with a freezing point below the lowest condenser temperature, see Chapter 3.5 "Technical data", page 19.

Damage or hazards attributable to use of the product other than as intended are entirely at the risk of the operator alone.

2.3 Staff qualification

Unqualified persons are unable to identify risks and are therefore exposed to greater dangers.

The instrument must be operated by suitably qualified laboratory staff. These operating instructions are aimed at the following target groups:

Users

The 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 must 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 mi- nor 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.





Location of the warning symbols on the instrument (front view)



Fig. 1: Location of warning symbols on the instrument (front)



Location of the warning symbols on the instrument (rear view)

Fig. 2: Location of warning symbols on the instrument (rear)

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 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 seals concerned and replace them if necessary.
- ▶ Do not process any unknown fluids.
- Observe the safety data sheets for all substances used.

2.7.3 Cold and hot surfaces

The condenser coil and probes can be extremely cold. Heatable shelf areas can be extremely hot. If touched, hot and cold surfaces can cause skin burns.

▶ Do not touch cold or hot surfaces or liquids and/or wear suitable protective gloves.

2.7.4 Glass and acrylic breakage

Broken glass and acrylic can cause severe cuts. Damaged glass and acrylic components may implode if subjected to a vacuum. Minor damage to the ground joints impairs the sealing effect and may therefore diminish sublimination capacity.

- Handle the flask and other glass and acrylic components carefully and do not drop them.
- Always place the flasks in a suitable holder when they are not mounted on the instrument.
- Always visually inspect glass and acrylic components for damage every time they are to be used.
- Do not continue to use glass and acrylic components that are damaged or in poor condition.
- ► Always wear protective gloves when disposing of broken glass and acrylic.

2.7.5 Damage to the ice condenser

Damage to the ice condenser leads to leakage of flammable refrigerant and failure of the instrument.

- ▶ Do not use mechanical means to remove ice from the ice condenser.
- ▶ Wait until the ice is completely thawed.
- ▶ Handle the ice condenser with care to avoid danger from flammable refrigerant.

2.7.6 Low internal pressure

Evacuating the system reduces the pressure in the drying chamber. This reduced pressure can cause glass and acrylic components to implode.

▶ Make sure that all glass and acrylic components are free of damage.

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.

3 Product description

3.1 Description of function

The instrument is a freeze-dryer in which frozen samples can be gently dried. The basis of freeze-drying is sublimation. Sublimation refers to the process whereby a substance transforms directly from the solid to the gaseous state.

The physical process of sublimation can be explained using the solvent water as an example.

- A solvent such as water in a sample is frozen.
- The frozen solvent is transformed into the gaseous state under vacuum at a pressure below the triple point.

Thus, freeze-drying takes place in three phases:

- 1. Freezing phase: the sample is frozen at atmospheric pressure.
- 2. **Main drying phase:** heat is applied to the frozen sample under a vacuum. The frozen water is removed by sublimation.
- 3. Secondary drying phase (only possible with heatable shelves): the trace levels of water remaining are removed by heating.

The instrument consists of an ice condenser and one choice of various top-mount drying racks. The top-mount drying racks can be chosen to suit the availability of the sample being dried and the requirements of the end product. The following top-mount drying racks can be used:

- Non-heatable and heatable shelves in rack
- Trays in rack
- Top-mount rack with manifold valves

3.1.1 Freezing phase

In the freezing phase, the aqueous preparation is transformed to a solid state. Freezing takes place under atmospheric pressure using a separate freezer, a liquid nitrogen bath, or a mixture of dry ice and alcohol.

The end of the freezing phase is reached as soon as the solvent contained in the preparation has fully crystallized.

3.1.2 Main drying phase

In the main drying phase, the ice crystals are removed from the preparation by sublimation. Sublimation in the instrument takes place under a vacuum with the addition of thermal energy.

To create the vacuum, the pressure is lowered to the level required for sublimation. As an example, for water that is less than 6.11 mbar.

As the ice condenser is colder than the preparation being dried, the vapour pressure in the area of the ice condenser is lower than in the area of the preparation. The solvent vapour escaping from the preparation therefore flows towards the ice condenser. The solvent vapour condenses on the coil of the ice condenser. If a manifold-valve rack is used, heat transfer takes place by convection and radiation from the surroundings. Control of the thermal energy transferred is then difficult. If using a drying chamber with heatable shelves, heat transfer takes place by direct contact. The temperature of the heatable shelves is controllable. Control of the thermal energy transferred is then possible. Controlling the heat transfer prevents the following critical temperatures for amorphous and crystalline materials to be reached:

- the glass transition temperature T_q' of the frozen preparation
- the collapse temperature T_c.
- the eutectic temperature T_{eu}.

Above the glass transition temperature and the collapse temperature, the viscosity of the frozen preparation increases. The increased viscosity leads to the collapse of the preparation's matrix structure.

Above the eutectic temperature, the preparation melts.

During the main drying phase, the product temperature must remain below the collapse temperature for amorphous materials in the preparation.

Sublimation of the ice crystals progresses downwards from the surface of the product. Above the sublimation boundary, the product is dry ("freeze-dried cake"), while further inside the product is still frozen.

The end of the main drying phase is reached when all ice crystals have been removed from the preparation.

After the main drying phase, the remaining fluid content in the preparation can still be between 5 to 10%.

3.1.3 Secondary-drying phase

In the secondary drying phase, the unfrozen solvent is removed from the sample by desorption. The secondary drying function is performed by the heatable shelves in the drying chamber of the instrument.

In the secondary drying phase, the temperature of the heatable shelves is raised and held for several hours.

The end of the secondary drying phase is reached when the residual moisture in the sample is between 1% and 5% or the end point determination is succeeded.

3.2 Configuration

3.2.1 Front view



Fig. 3: Front view

- Connections for heated shelves (Lyovapor[™] L-250 Pro only) (48 V, max. 2 A)
- 3 Ventilation slots
- 5 Control panel

- 2 Ice condenser
- 4 On/Off master switch

3.2.2 Rear view



Fig. 4: Rear view

- 1 Connections on the rear side 2 Drain valve (See Chapter 3.2.3 "Connections on the rear side", page 17)
- 3 Ventilation slots





- Vacuum pump connection (200 - 240 VAC, 550 W)
- 3 Type plate
- 5 USB 2.0 port
- Pressure regulation valve connection 8 7 (24 V, ≤ 0.4 A)
- 9 External vacuum sensor connection (24 V, max. 0.125 A)
- 11 LAN connection
- 13 Power supply connection

- Refrigerant specification 4
- Main valve connection 6 $(24 \text{ V}, \le 0.4 \text{ A})$
 - Aeration valve connection (24 V, ≤ 0.4 A)
- 10 Vacuum sensor connection (5 V, max. 0.125 A)
- 12 COM port
 - (24 V, ≤ 0.4 A)
- 14 Fuses

3.2.4 Control panel



Fig. 6: Control panel

- 1 Touch screen display
- 2 Navigation control

3.3 Scope of delivery

NOTE

i

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 Type plate

The type plate identifies the instrument. The following type plate is an example. For more details refer to the type plate on the instrument. The type plate is located at the rear of the instrument.



2

4

6

8

Instrument name

Product origin

sessed"

(optional)

household waste"

Input voltage range

Power consumption maximum

10 Symbol for "Do not dispose of as

12 Symbol for "UK Conformity As-

14 Symbol for "Eurasian Conformity"

- 1 Company name and address
- 3 Serial number
- 5 Frequency
- 7 Year of manufacture
- 9 Symbol for "CE conformity"
- 11 Symbol for "electronics recycling"
- 13 QR-Code contains "Item number, Serial number"
- 15 Symbol for "CSA certified" (optional)

3.5 Technical data

3.5.1 Lyovapor™ L-250 Basic/L-250 Pro

Specification	L-250 Basic	L-250 Pro
Dimensions without drying attach- ments (W x D x H)	503 x 645 x 510 mm	503 x 645 x 510 mm
Weight	65.6 kg	67.4 kg
Minimum clearance on all sides	300 mm	300 mm
Connection voltage	200 – 240 VAC ±10%	200 – 240 VAC ±10%
Rated power consumption	1,300 VA	1,800 VA
Fuse	10 A	10 A
Frequency	50 / 60 Hz	50 / 60 Hz
Overvoltage category	II	
Protection rating	IP20	IP20
Pollution degree	2	2
Condensing capacity	4 kg / 24 h	4 kg / 24 h
Lowest condenser temperature	-85 °C	-85 °C
Temperature divergence	±1.0 °C	±1.0 °C
Condenser capacity	≤ 5 kg	≤ 5 kg
Condenser surface area	1,524 cm ²	1,524 cm ²
Refrigerants	R290, R600, R1150	R290, R600, R1150
Refrigerant quantity	< 100 g	< 100 g

Specification	L-250 Basic	L-250 Pro
Vacuum generation time to 0.1 mbar	Typically ≤ 10 min	Typically ≤ 10 min
Volume-based leakage rate	< 10 mbar L/h	< 10 mbar L/h
Lowest system vacuum	0.03 mbar	0.03 mbar
Control range vacuum	0.1 – 1 mbar	0.1 – 1 mbar
Shelf heating	max. 60 °C	max. 60 °C
Shelf temperature control tolerance	±1.0 °C	±1.0 °C
Certificates	CE/CSA/CB	CE/CSA/CB
Drying attachments operating height	1,100 mm	1,100 mm
Noise emission to DIN 45635 (without vacuum pump)	< 60 dB(A)	< 60 dB(A)
Inert gas connection (relative)	0.5 bar	0.5 bar
Marking on type plate	Bas / BasD	Pro / ProD
Defrosting (optional, D on type plate)	Yes	Yes

3.5.2 Ambient conditions

For indoor use only.

Max. altitude above sea level	2'000 m
Ambient temperature	5 - 30 °C ¹
Maximum relative humidity	80 % for temperatures up to 30 °C
Storage temperature	max. 70 °C

¹ Above 30°C the ice condenser power decreases. Reduce sample quantity to prevent sample collapse.

3.5.3 Materials



NOTICE

Material damage from use of organic solvents and acids.

Use of organic solvents and acids can wear out components made of PMMA over time.

- ► Consider the material compatibility towards solvents, see document *List of chemical resistances Lyovapor*[™].
- Regularly check PMMA components.

Housing	Steel 1.4301/304 with powder coating
Vacuum chamber and components	Steel 1.4404
Main connector	PE-UHMW 1000
Drying chamber tube and cover	PMMA GS
General seals	FKM
Drain valve seals	PTFE/EPDM
Manifold drying rack	Steel 1.4301/304
Manifold valves	EPDM, PP
Cooling medium circuit	Copper for freezing applications to EN 12735-1
Vacuum clips	Aluminum
Condensate drain tube	Steel 1.4301/304/Silicone

Ventilation	Brass with EPDM drain valve seals
Main valve, control valve	Brass with FKM seal
Condensate valve	Steel with EPDM seals

Used instrument materials such as steel and packaging originate from recycling processes.

3.5.4 Installation site

The installation site must meet the following requirements:

- The installation site has a firm, level surface.
- The installation site has a fume hood.
- The installation site allows that the power supply can be disconnected at any time in an emergency.
- The installation site has enough space that cables / tubes can be routed safely.
- The installation site meets the requirements for the connected devices. See related documentation
- There is no loose paper or the like at the installation site that could drawn in the ventilation.
- The installation site meets the specifications according to the technical data (e.g. weight, dimension, etc.). See Chapter 3.5 "Technical data", page 19
- The installation site is not exposed to external thermal loads, such as direct solar radiation.
- The installation site meets the safety requirements. See Chapter 2.2 "Use other than intended", page 8.
- Make sure that the feet do not bend when installing the instrument.
- When mounting on an instrument cart, the feet of the instrument must be placed into the supports of the instrument cart.
- The installation site has enough space for the operating height of the drying attachments, see Chapter 3.5 "Technical data", page 19.
- The installation site allows for a min. clearance of 300 mm on each side of the instrument. This clearance ensures air circulation and prevents the instrument from overheating.

3.6 Refrigerant specification

The instrument uses a refrigerant mixture to maintain the ice condenser temperature. For details see Chapter 3.5 "Technical data", page 19.



- Fig. 7: Refrigerant details
- 1 Instrument name
- 3 Filling capacity

- 2 Refrigerant details
- 4 Design pressure

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 3.5 "Technical data", page 19).
- ▶ Wherever possible, store the instrument in its original packaging.
- After storage, check the instrument, 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.



Fig. 8: Lifting the instrument

- 1 Instrument
 - Equipment trolley
- 2 Feet
- 4 Castor brakes on trolley

Precondition:

3

- $\ensuremath{\boxtimes}$ Make sure that the castor brakes on the equipment trolley are locked on.
- Lift the instrument this requires two persons each lifting at two of the points indicated on the front and rear of the instrument.
- ► Set the instrument down on the equipment trolley.

5 Installation

5.1 Before installation



Risk of explosion from flammable gas-and-air mixtures

The potential consequences are death or extremely serious injury.

- ▶ Do not damage the refrigerant circuit piping.
- Store and operate the instrument in a room with a volume of at least 16.6 m³ so that an ignitable gas-and-air mixture cannot develop.



NOTICE

Product loss from power failure and unstable power supply.

Power failures disrupt the operation and cause product loss.

When working with expensive samples or if the power supply is unstable, use an Uninterruptible Power Supply.

5.2 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.



NOTICE

The power supply cable is the disconnecting device.

Easy access must be guarantee to the mains plug at all times.

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 data. See Chapter 3.5 "Technical data", page 19.
- Connect the power supply cable to the connection on the instrument. See Chapter 3.2 "Configuration", page 15.



 Connect the mains plug to an own mains outlet socket.

5.3 Securing against earthquakes

The instrument has an earthquake fixing point to protect the instrument against falling.



Fig. 9: Fixation hole

- 1 Fixation hole
- ▶ Tie a steel rope to the fixation hole to secure the instrument.

5.4 Putting the instrument in operation



NOTICE

Instrument damaged if restarted too early

Wait ten minutes before restarting the instrument. The oil in the refrigerant compressor requires ten minutes to return to the collection tank.

5.4.1 Preparing the instrument

- ► Clean the instrument with a damp cloth before commissioning.
- ► Check all sealing surfaces for scratches, dust, and cleanliness.

5.4.2 Fitting the advanced vacuum control unit

▶ Fit the 90° elbows onto the cross-pipe.



► Fit the pressure regulation valve onto the 90° elbow.



Option 1

- ► Attach the main valve to the cross pipe.
- ▶ Fit the vacuum tubing to the main valve.
- ▶ Skip the next two steps to proceed.





Precondition:

- \boxdot The arrow is pointing downward.
- ► Fit the main valve onto the cross-pipe.

 Connect the tubing vertically to the 90° elbow.





5.4.3 Fitting the ultimate vacuum control unit

► Fit the vacuum tubing onto the 90° elbow.



► Attach the 90° elbow to the vacuum connection on the instrument.



5.4.4 Connecting the inert gas (optional)

Precondition:

- ☑ Make sure that the pressure of the inert gas matches the technical specifications. See Chapter 3.5 "Technical data", page 19.
- Fit the inert gas tube onto the gas connection on the pressure regulation valve and regulation valve.
- ► Fix the inert gas tube with the tube clip.

5.4.5 Assembling pressure sensor PPG011

The pressure sensor measures the pressure in the ice condenser. To protect against damage in transit, the pressure sensor is supplied pre-calibrated in the original packaging.





Fig. 10: Assembling pressure sensor PPG011

- 1 Pressure sensor PPG011 2 Clamp, ISO-KF 16
- 3 Seal with sintered filters, ISO-KF 16 4 Connection, ISO-KF 16
- ► Switch the On/Off master switch to Off.
- ▶ Remove the shipping cap from the connection (4).
- ► Fit the pressure sensor (1) and seal (3) to the connection (4) and fix with the clamp (2).
- Plug the pressure sensor electrical connection into the socket marked Vacuum Sensor.
- ▶ Select the sensor from the submenu [Settings] on the control panel.

5.5 Commissioning the vacuum pump

The vacuum pump evacuates the top-mount drying rack during the freeze-drying process.



\Lambda DANGER

Risk of electric shock due to high leakage currents.

Serious injuries or death can result.

- No additional earth connection is needed for safe operation of the following vacuum pumps:
 - Edwards nXDS6iC
 - Pfeiffer DUO 6M
- If a different vacuum pump is used, which must be a third party tested and NRTL certified type, an additional earthing connection is needed.
- Additional earthing connection must be installed by a qualified electrician between the earthing terminal provided on the vacuum pump and a safe on-site earth.



NOTICE

Open gas ballast valve.

A closed gas ballast valve when using solvents can cause damage to the instrument.

▶ Open the gas ballast valve.



NOTE

The equipment must be installed by trained staff.



NOTE

To increase the service life of the vacuum pump, operate the vacuum pump with an open gas ballast valve.



NOTE

Prepare the vacuum pump according to the manufacturer's instructions. See relevant documentation.



Fig. 11:

1 Vacuum pump power connection

- 2 Vacuum tube connection, ISO-KF 25
- Switch the **On/Off** master switch to Off.
- ► Connect the vacuum pump vacuum tube to the vacuum tube connection (2).
- Plug the vacuum pump electrical connection into the socket marked Vacuum Pump.

5.6 Establishing LAN connection

5.6.1 Requirements for local network settings

- The following port has to be enabled in the firewall settings on the internet gateway:
- TCP (HTTPS) traffics through remote port 443
- In order to use the BUCHI Cloud a DNS server must be configured on the instrument.



NOTE

If there is no DNS server available enter the IP address for the BUCHI Cloud connection manually.



NOTE

If there is no DHCP server available enter the IP address, gateway subnet mask and DNS server manually.

5.6.2 Preparing the instrument for app using

NOTICE! Do not unplug the LAN cable while the unit is connected to the BUCHI Cloud Services.

- Connect the unit to the LAN (local area network).
- Restart the unit.



Navigation path

 \rightarrow ^(☉) \rightarrow [Settings] \rightarrow [Network]

- ► Navigate to [Network].
- Activate the function [DHCP].
- \Rightarrow The unit is now ready.

5.6.3 Enabling BUCHI Cloud access

Enable access to BUCHI Cloud in order to use the BUCHI Monitor App.

Navigation path

- → \bigotimes^{3} → [Settings] → [Network] → [BUCHI Cloud]
- ▶ Navigate to the action [BUCHI Cloud] via the navigation path.
- Select the option [Yes].
- \Rightarrow The instrument is connected to the BUCHI Cloud.

5.7 Inserting the SD card



NOTE

Only insert or remove the SD card in Standby mode and in Power off mode

► Fold the control panel forward.



▶ Insert the SD card on the underside.



- Switch on the instrument.
- ⇒ The status bar shows the SD card symbol.

The following data is stored on the SD card:

- Numbering
- Date
- Time
- Set pressure
- Current pressure in the ice condenser
- Inlet temperature of the ice condensers
- Set drying shelf temperature
- Current temperature of the drying shelves
- Current sample temperatures

5.8 Installing the vacuum pump tray



Risk of the trolley tipping over

- ▶ The vacuum pump must not weigh more than 30 kg.
- The main device must stand on the trolley when the vacuum pump is placed on the tray.
- Guide the tray at an angle to the metal strut on the trolley side where there are no brakes on the wheels.



Turn the tray downwards until it is in a horizontal position.





6 Interface

6.1 Layout of the control panel



Fig. 12: Layout of the control panel

Description	Function
Status bar	Shows the current status of the instrument.
Menu bar	Shows symbols representing the menus.
Content area	Shows current settings, submenus or actions depending on the current operation.
Function bar	Shows functions that can be performed ac- cording to the current operation
Navigation control	Used for navigating the user interface. Press- ing the control performs the assigned function on the function bar.
	DescriptionStatus barMenu barContent areaFunction barNavigation control

6.2 Function bar

The function bar shows functions that can be performed according to the current operation.

The functions on the function bar are executed by tapping the relevant function buttons or pressing the navigation control.

General function buttons

Symbol	Description	Meaning
	[Back]	The display reverts to the previous view.
ESC	[Cancel]	Cancels an operation.
→ ☆	[Add to favourites]	Adds the selected item to the <i>[Favourites]</i> menu.
OK	[Confirm]	Confirms an entry.
EDIT	[Edit]	Allows the selected item to be edited.
MENU	[Menu]	Allows selection of a menu from the menu bar using the navigation con- trol.

Symbol	Description	Meaning	
SAVE	[Save]	Saves the setting.	
Process control function buttons			
Symbol	Description	Meaning	
AERATE	[Aerate]	Vents the system.	
🔆 OFF	[Shut down]	The instrument shuts down.	
START	[Start]	Starts the freeze-drying process.	
+∰ ON	[Start conditioning]	Starts the conditioning phase.	
MANUAL	[Manual]	Switches to manual freeze-drying.	
METHOD	[Method]	Switches to freeze-drying with pro- grammable parameters.	
NEW	[New]	Creates a new method	
>	[Right]	Moves the selection to the right.	
<	[Left]	Moves the selection to the left.	
m	[Progression]	Graphical display of method progres- sion showing pressure and tempera- ture details.	
ACTIVATE	[Activate]	Confirms selection of a method.	
DELETE	[Delete]	Deletes the selected entry.	
SKIP	[Skip]	Skips the current process.	
UNPROT	[[Deactivate sample protection]]	Manually deactivates sample protec- tion.	
COPY	[Copy]	Copies the selected method.	
OPEN	[Open]	Opens the selected valve.	
CLOSE	[Close]	Closes the selected valve.	
⊘ ON	[Vacuum pump ON]	Switches on the vacuum pump.	
Ø OFF	[Vacuum pump OFF]	Switches off the vacuum pump.	
∰ ON	[Start defrost]	Starts the defrosting of the ice con- denser.	
+¥ OFF	[Stop defrost]	Stops the defrosting of the ice con- denser.	
(l ²	[Extend time]	Extends the defrosting by 30 min.	
<u>1</u>	[Reduce time]	Reduces defrosting time by 30 min.	
6.3 Other symbols on the control panel

Symbol	Description	Meaning
T	[Closed]	The assigned method is active and cannot be changed.

6.4 Menu bar

The menus are represented by symbols on the menu bar. Navigation through the menus is by the input controls.

The following menus are available:

Menu symbol	Meaning	Submenu/Action
\bigcirc	[Start] menu	 Process control parameters
\sum	<i>[Favourites]</i> menu	 Bookmarks for individual entry points
\sim	[Method] menu	For saving freeze-drying methods
		• Editing and activating freeze-drying method
$\langle \dot{\gamma} \rangle$	[Configuration] menu	Process settings
		Settings
	-	 End point determination
		Maintenance
		Service
		System information
	[Messages] menu	Notifications
		• Journal
$\sqrt{1}$	[Graph] menu	Live graph displaying the current:
Í		 Ice condenser temperature
	_	 Ice condenser pressure
		Sample temperature
		Shelve temperature

6.4.1 Start menu

On the [Start] menu, parameters can be set manually.

Setting parameters using the navigation control

- ▶ Select a parameter by turning the navigation control.
- \Rightarrow The control panel highlights the selected parameter in green.
- ▶ Tap the function [Edit] on the function bar.
- \Rightarrow The control panel highlights the selected parameter in black.
- ▶ To increase or decrease the figure, use the dialog box with a numeric input.
- Press the navigation control.
- \Rightarrow The setting is saved.
- \Rightarrow The control panel highlights the new setting in green.

Setting parameters using the touch-screen

- ▶ Select the parameter by tapping the control panel screen.
- \Rightarrow The control panel shows a dialog box with a numeric input box.
- \Rightarrow The control panel highlights the selected parameter in black.
- Enter the value in the numeric input box.
- ► Tap the function [Save] on the function bar.
- \Rightarrow The setting is saved.
- \Rightarrow The dialog box closes.
- \Rightarrow The control panel highlights the new setting in green.

6.4.2 Favourites menu

The [Favourites] menu allows you to define submenus and actions as favourites.

Adding a favourite

- ▶ Navigate to a submenu or action.
- ► Tap the function [Add to favourites] on the function bar.
- ⇒ The user interface switches to the [Favourites] menu and displays the favourite created.

Removing a favourite

- ▶ On the *[Favourites]* menu, navigate to the favourite you wish to remove.
- ► Tap the function *[Delete]* on the function bar.
- \Rightarrow The favourite is removed.

6.4.3 Method menu

The *[Method]* menu allows freeze-drying processes with multiple phases and steps to be saved. See Chapter 7.2 "Editing a method", page 44.

6.4.4 Configuration menu

On the *[Configuration]* menu, you can enter a variety of settings and retrieve information.

6.4.5 Messages menu

The *[Messages]* menu shows the current instrument messages and the instrument's message history.

The following message types are possible:

- I = Information: No immediate actions by customer are needed.
- W = Warning: Minor failures during operation. Customer action is required.
- E = Error: Major failures during operation due to defective system component. Usually service support is needed.

6.4.6 Graph menu

The *[Graph]* menu allows viewing the current freeze drying process via a live graph. The live graph displays the following parameters:

- Ice condenser temperature
- Ice condenser pressure
- Sample temperature
- Shelve temperature

6.5 Status bar

The status bar shows the status of the instrument.

The following statuses are possible:	
Indication on status bar	
Unload / Load	Conditioning is completed.
	Before the freeze-drying process:
	Load the top-mount drying rack with a frozen sample.
	After the freeze-drying process:
	Remove the dried sample from the top- mount drying rack.
Aerating	The system is venting.
Shutting down	The instrument is shutting down.
	Place a container underneath the drain valve.
	Open the drain valve manually to drain the melted ice from the ice condenser. • The aeration valve is closed.
	 The status bar shows the remaining time.
Defrosting	The instrument is defrosting.
	Place a container underneath the drain valve.
	Open the drain valve manually to drain the melted ice from the ice condenser.
	 The aeration valve is open. The status bar shows the remaining time.
Standby	Shutting down is completed.
Conditioning	The instrument is starting up including starting refrigerant compressor and vac- uum pump.
	Close the drain valve manually.
Reconditioning	The instrument is restarting after a tempo- rary power failure (< 15 min).
Warming up pump	The vacuum pump is being brought up to operating temperature.
Vacuum Test	The instrument is performing a vacuum test.
Leak Test	The instrument is performing a leak test.
Manual Drying	The instrument is in the course of a man- ual freeze-drying process.
Recovering	The system is in the process of recover- ing from a power failure (> 15 min).
	The current parameters of the freeze-dry- ing process are being re-established.
Hold	The instrument is in the holding phase.
Primary drying	The instrument is in the primary drying phase.

Secondary drying	The instrument is in the secondary drying phase.
Tempering shelves	The instrument is modulating the heatable shelves to the set temperature.
Stoppering	The instrument is ready for stoppering.

Symbols on the status bar

Symbol	Status
÷¥.	The instrument is defrosting.
0	The instrument is in energy-saving mode.
	The instrument is in the course of a freeze-drying process using a method.
***	The instrument is starting up.
₩	The instrument is in the course of a manual freeze-drying process.
	The instrument is connected to the BUCHI Cloud.
P ev	Sample protection is active.
	Reason: the pressure is outside the pressure lim- its.
T ev	Sample protection is active.
	Reason: the temperature is outside the safe tem- perature range.
PT	Sample protection is active.
	Reasons:
	The pressure is outside the pressure limits.
	The temperature is outside the safe temperature range.
<u>↓↑</u>	Before the freeze-drying process:
	Load the top-mount drying rack with a frozen sample.
	After the freeze-drying process:
	Remove the dried sample from the top-mount dry- ing rack.
Т	The instrument is performing a vacuum test or a leak test.
V	The system evacuates to the set pressure.
SD	The memory card has been inserted.

7 Operation



Cutting hazard from broken glass.

Broken flasks can cause cuts.

- Check the flasks for any damages, cracks or scratches before each use.
- ▶ Do not use any flasks that are not in optimal condition.
- ► Handle the flasks carefully.



NOTICE

Instrument damage from glass splinters.

Sharp objects can damage the display.

▶ Keep sharp objects away from the display.



NOTICE

Instrument damage from spilled liquids.

Liquid substances can cause stains and damage the instrument.

Immediately wipe away any spilled liquids.



NOTE

Empty the ice condenser before operation.

Processing does not work if the ice condenser is not empty before operation.

• Empty the ice condenser before processing.

7.1 Performing freeze-drying manually

7.1.1 Preparing the instrument

Time required:

approx. 30 min



NOTE

To minimize the condensation of moisture from the air in the condenser, place a drying rack. Remove any residual moisture from the ice condenser and close the drain valve.

Navigation path

→ [Start]

- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function [Start conditioning] on the function bar.
- \Rightarrow The temperature in the ice condenser decreases to operating temperature.
- \Rightarrow The vacuum pump is brought up to operating temperature.
- After completion of the conditioning phase, the status bar shows the status Unload / Load.

Preparing the instrument with ultimate vacuum control unit

Time required:

approx. 45 min

Navigation path

→[Start]

- Make sure, that the ice condenser is dry and the drain valve is closed.
- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function [Start conditioning] on the function bar.
- ► Install the manifold drying rack.
- ► As soon as the ice condenser temperature is reached, close the valves.
- ► Tap the [Vacuum pump ON] function on the function bar.
- \Rightarrow The vacuum pump switches on.
- Consider the warm-up time manually.

7.1.2 Starting freeze-drying



Risk of skin burns from touching parts of the ice condenser after completion of conditioning.

 Wear protective gloves when working on the instrument after the conditioning phase.

Navigation path

→ [Start]

Precondition:

 \boxdot The instrument has been prepared.

- ► Fit a top-mount drying rack. See Chapter 7.9 "Operating top-mount drying racks", page 57.
- ► Load the top-mount drying rack with frozen samples.
- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function [Manual] on the function bar.
- ► Enter the required settings for the process parameters.
- ► Tap the function [Start] on the function bar.
- ⇒ The freeze-drying process starts.
- ⇒ The background color of the Start menu changes from white to black.
- \Rightarrow The status bar shows a clock counting up and the status **Manual Drying**.
- \Rightarrow The system evacuates to the set pressure.

7.1.3 Editing parameters while the process is running

Navigation path

→ [Start]

Precondition:

 \boxdot The process has been started.

- ▶ Navigate to the [Start] menu via the navigation path.
- ▶ Using the navigation control, navigate to the parameter that you wish to change.
- ► Tap the function [Edit] on the function bar.
- \Rightarrow The control panel shows a dialog box with a numeric input box.
- \Rightarrow The control panel highlights the selected parameter in white.

- Enter the value in the numeric input box.
- ► Tap the function *[Save]* on the function bar.
- \Rightarrow The setting is saved.
- \Rightarrow The dialog box closes.

7.1.4 Performing a pressure difference test for manifold drying rack



NOTE

The pressure difference test can only be carried out with a pressure difference test kit. See Chapter 11.1.1 "Accessories", page 88.

The pressure difference test establishes the difference between the readings from two pressure sensors in the drying chamber. If the difference between the two sensor readings is below a threshold, the freeze-drying phase can be ended.

→ [Configuration] → [End point definition]		
Setting	Option	Explanation
[Limit]	Enter value	Specifies the difference between the two sensor readings below which the end point is reached.
		The threshold must be greater than the offset value from the vacuum test. See Chapter 7.4.3 "Determining offset value", page 52
[Time]	Enter value	Specifies the length of time for which the pressure difference test is to be carried out until the endpoint is reached.
[Message]	Yes/No	A message that is displayed as soon as the pressure difference test is passed.

Navigation path

Precondition:

- ☑ A vacuum test has been carried out with a pressure equal to the pressure during the subsequent freeze-drying process. See Chapter 8.14 "Performing a vacuum test", page 78
- ☑ The sensors for the pressure difference test have been fitted. See "Connecting the sensors for a pressure difference test"
- ☑ The offset value has been determined. See Chapter 7.4.3 "Determining offset value", page 52
- \boxdot All flasks have been attached to the manifold drying rack.
- ▶ Navigate to [End point definition] via the navigation path.
- ▶ Tap [Pressure difference test].
- Set up the pressure difference test as desired.
- Tap [Start] to start the pressure difference test.

7.1.5 Ending freeze-drying

Navigation path

→ Start

Precondition:

 \boxdot The sample is dry.

- ▶ Navigate to the [Start] menu via the navigation path.
- ▶ Tap the function [Aerate] on the function bar.
- Answer **YES** to the confirmation question.
- \Rightarrow The system is vented.
- \Rightarrow The status bar shows the status **Aerating**.
- As soon as the status bar shows the status Unload / Load, remove the dried sample from the drying rack.

Ending freeze drying with ultimate vacuum control unit

Navigation path

→ Start

Precondition:

 \boxdot The sample is dry.

- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function *[Aerate]* on the function bar.
- ► Answer **YES** to the confirmation question.
- \Rightarrow The system is vented.
- \Rightarrow The status bar shows the status **Aerating**.
- ► As soon as the status bar shows the status **Unload** / **Load**, remove the flasks.
- ► Tap the [Vacuum pump OFF] function on the function bar.
- \Rightarrow The vacuum pump switches off.

7.2 Editing a method

The control panel can save up to 35 methods. The methods enable the freeze-drying process to be automated.

7.2.1 Creating a new method

There are two possible ways of creating a new method:

Creating a new method

Navigation path

→ [Method]

- ▶ Navigate to the [Method] menu via the navigation path.
- ► Tap the function *[New]* on the function bar.
- \Rightarrow The new method is created.

Creating a new method by copying an existing method

Navigation path

→ [Method]

- Navigate to the [Method] menu via the navigation path.
- Tap the name of the method that you wish to copy.
- ► Tap the function [Copy] on the function bar.
- \Rightarrow The new method is created.

7.2.2 Changing the name of a method

Navigation path

→ [Method]

- ▶ Navigate to the *[Method]* menu via the navigation path.
- ▶ Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ▶ Tap the action [Information].
- \Rightarrow The control panel shows the action Information.
- ► Tap the setting [Name].
- \Rightarrow The control panel shows a blank box with an alphanumeric input box.
- Enter a name for the method.
- ► Tap the function [Save] on the function bar.
- \Rightarrow The new name is saved.
- \Rightarrow The dialog box closes.

7.2.3 Setting the sample collapse temperature

Navigation path

→ [Method]

- ▶ Navigate to the *[Method]* menu via the navigation path.
- Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ▶ Tap the action [General].
- \Rightarrow The control panel shows the action [General].
- ► Tap the setting [Sample collapse temperature].
- \Rightarrow The control panel shows a dialog box with a numeric input box.
- Enter the value in the numeric input box.
- Tap the function [Save] on the function bar.
- \Rightarrow The setting is saved.
- \Rightarrow The dialog box closes.

7.2.4 Setting the gas type

Navigation path

→ [Method]

- ▶ Navigate to the *[Method]* menu via the navigation path.
- ► Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [General].
- \Rightarrow The control panel shows the action [General].
- ► Tap the setting [Gas type].
- \Rightarrow The control panel shows a dialog box with an alphanumeric input box.
- Enter the gas type.
- ▶ Tap the function [Save] on the function bar.
- \Rightarrow The setting is saved.
- \Rightarrow The dialog box closes.

7.2.5 Setting the shelf loading temperature

Navigation path

→ [Method]

- ▶ Navigate to the [Method] menu via the navigation path.
- ▶ Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [General].
- \Rightarrow The control panel shows the action [General].
- ► Tap the action [Shelf load temp.]
- $\Rightarrow\,$ The control panel shows a dialog box with a numeric input box.
- Enter the value in the numeric input box.
- ► Tap the function [Save] on the function bar.
- \Rightarrow The setting is saved.
- \Rightarrow The dialog box closes.

7.2.6 Setting the steps of a method

The control panel can save up to 30 steps for each method.

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NOTE

The maximum heating rate is 3 °C/min.

NOTE

The settings for the action steps affect a single step in each case.

Navigation path

→ [Method]

- ▶ Navigate to the [Method] menu via the navigation path.
- Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [Steps].
- \Rightarrow The control panel shows the action Steps.

The following settings are available for each step:

Setting	Option	Meaning
[Step phase]	Primary drying/Sec- ondary drying	Sets the type of step phase.
[Term]	Enter value	Sets the duration of the step in minutes.
[Shelf temperature]	Enter value	Set the temperature of the heat- able shelves in one step.
[Pressure zone]	Regulated/Minimum	Regulated: the settings for pres- sure and pressure limits are ap- plied.
		Minimum: The maximum vacuum is applied to reach the lowest possible pressure.
[Pressure]	Enter value	Sets a target value for the regulated pressure.

Setting	Option	Meaning
[Pressure limit]	Enter value	Absolute value for divergence from the set pressure before the sample protection function is acti- vated.
[Pressure duration]	Enter value	Sets the period of time that the pressure is allowed to exceed the pressure limit before the sample protection function is activated.

Editing a step

- ► Use the function [*Right*] or [*Left*] on the function bar to navigate to the step that you wish to edit.
- ▶ Using the navigation control, navigate to the setting that you wish to change.
- ► Tap the function *[Edit]* on the function bar.
- ► Edit the setting as required.
- ► Tap the function [Save] on the function bar.
- \Rightarrow The setting is changed.

Add step

- ► Use the function [*Right*] or [*Left*] on the function bar to navigate to the position at which you wish to add a step.
- ► Tap the function *[New]* on the function bar.
- \Rightarrow The new step is created.

Delete step

- ► Use the function [*Right*] or [*Left*] on the function bar to navigate to the step that you wish to delete.
- ► Tap the function [Delete] on the function bar.
- ▶ When asked to confirm, press [OK].
- \Rightarrow The step is deleted.

7.2.7 Setting the phases of a method

NOTE

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The settings in the Phase view affect all steps of a phase.

Navigation path

→ [Method]

- ▶ Navigate to the [Method] menu via the navigation path.
- ▶ Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [Phase].
- \Rightarrow The control panel shows the Phase view.

The following phases of a method are available:

Phase	Setting	Option	Meaning
[Primary drying]	[Pressure ac- tion]	None/Sample protection/Mes- sage	None: no action is carried out.
			Sample protection: if the pressure is too high, heating of the shelves is paused.
			Message: if the pressure is too high, the control panel displays a message.
	[Temp. action]	None/Sample	None: no action is carried out.
		protection/Mes- sage	Sample protection: if the temperature is too high, heating of the shelves is paused.
			Message: if the temperature is too high, the control panel displays a message.
	[Safety tem- perature]	Enter value	Maximum divergence from the set collapse temperature before the sample protection function is activated.
	[Safety temp. Time]	Enter value	Time as of which sample protection is inactive. The value relates to the time before completion of primary drying.
	[End point def- inition]	More settings	See Chapter 7.4 "Setting end point definitions", page 49
[Sec- ondary drying]	[Pressure ac- tion]	None/Sample protection/Mes- sage	None: no action is carried out.
			Sample protection: if the pressure is too high, heating of the shelves is paused.
			Message: if the pressure is too high, the control panel displays a message.
	[Temp. action]	None/Sample protection/Mes- sage	None: no action is carried out.
			Sample protection: if the temperature is too high, heating of the shelves is paused.
			Message: if the temperature is too high, the control panel displays a message.
	[Safety tem- perature]	Enter value	Maximum divergence from the set shelf temperature before the sample protection function is activated.
	[End point def- inition]	More settings	See Chapter 7.4 "Setting end point definitions", page 49
[Stopper- ing]	[Pressure zone]	Regulated/Mini- mum	Regulated: the settings for pressure and pressure limits are applied.
			Minimum: The maximum vacuum is applied to reach the lowest possible pressure.
	[Pressure]	Enter value	Sets a target value for the regulated pressure.
	[&Mode]	None/Manual	None: no action is carried out.
			Manual: sealing is performed manu- ally.

Phase	Setting	Option	Meaning
[hold]	[Pressure zone]	Regulated/Mini- mum	Regulated: the settings for pressure are applied.
			Minimum: The maximum vacuum is applied to reach the lowest possible pressure.
	[Pressure]	Enter value	Sets a target value for the regulated pressure.
	[Shelf temper- ature]	Enter value	Specifies a temperature for the shelves.

Editing the settings for a phase

- ▶ Tap the phase that you wish to edit.
- \Rightarrow The control panel highlights the selected phase in green.
- ► Tap the setting that you wish to edit.
- ► Edit the setting as required.
- ► Tap the function *[Save]* on the function bar.
- \Rightarrow The setting is changed.

7.3 Deleting a method

Navigation path

→ [Method]

- ▶ Navigate to the *[Method]* menu via the navigation path.
- ► Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the function [Delete] on the function bar.
- Select [Confirm] to confirm the action in response to the confirmation question.
- \Rightarrow The method is deleted.

7.4 Setting end point definitions

The end of a phase can be automatically set for each preparation by defining an end point.

The end point can be defined by means of the temperature difference test or the pressure difference test.

7.4.1 Performing a pressure difference test (option)

Connecting the sensors for pressure difference test

- ► Fit the pressure sensor, seal to the connection and fix with the clamp.
- Plug the pressure sensor electrical connector into the socket marked Vacuum Sensor.



- ► Fit the pressure sensor, seal to the connection and fix with the clamp.
- Plug the pressure sensor electrical connector into the socket marked External Vacuum Sensor.
- ► On the control panel, select the sensor Inficon Porter CDG020 D on the [Settings] submenu.



For manifold drying rack:

► Fit the manifold drying rack onto the sensor.



Performing a pressure difference test for drying chamber

i

NOTE

The pressure difference test can only be carried out with a pressure difference test kit. See Chapter 11.1.1 "Accessories", page 88.

The pressure difference test establishes the difference between the readings from two pressure sensors in the drying chamber. If the difference between the two sensor readings is below a threshold, the freeze-drying phase can be ended.

Navigation path

→ [Method]

Precondition:

- ☑ A vacuum test has been carried out with a pressure equal to the pressure during the subsequent freeze-drying process. See Chapter 8.14 "Performing a vacuum test", page 78
- ☑ The sensors for the pressure difference test have been fitted. See Chapter "Connecting the sensors for pressure difference test", page 49
- ☑ The offset value has been determined. See Chapter 7.4.3 "Determining offset value", page 52
- ▶ Navigate to the [Method] menu via the navigation path.
- ► Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [Phase].
- \Rightarrow The control panel shows the action Phase.
- ► Tap the setting [End point definition].
- \Rightarrow The control panel shows the setting End point definition.

- ▶ Tap [Pressure difference test].
- \Rightarrow The control panel shows the pressure difference test.

The following settings are available:

Setting	Option	Explanation
[Pressure dif- ference test]	Yes/No	Switches the pressure difference test on or off.
[Start time]	Enter value	Sets the time from which the pressure difference test is to be performed. The value relates to the time before completion of the primary drying phase.
[Pressure dif- ference limit]	Enter value	Specifies the difference between the two sensor readings below which the end point is reached.
		The threshold must be greater than the offset value from the vacuum test. See Chapter 7.4.3 "Determining offset value", page 52
[Term]	Enter value	Specifies the length of time for which the pressure difference test is to be carried out.
[Continue]	Yes/No	Yes : the method switches to the next phase.
		No: the phase is ended when the set levels are reached.
[Message]	Yes/No	The control panel shows or does not show a message as soon as the pressure difference test is passed.

7.4.2 Performing a pressure difference test for manifold drying rack (option)



NOTE

The pressure difference test can only be carried out with a pressure difference test kit. See Chapter 11.1.1 "Accessories", page 88.

The pressure difference test establishes the difference between the readings from two pressure sensors in the drying chamber. If the difference between the two sensor readings is below a threshold, the freeze-drying phase can be ended.

Connecting the sensors for a pressure difference test

- ▶ Fit the manifold drying rack onto the sensor kit (order no. 11080770).
- Mount the sensor kit on top of the manifold drying rack.

Navigation path

→ [Configuration] → [End point definition]

The following settings are available:

Setting	etting Option Explanation		
[Pressure dif- ference limit]	Enter value	Specifies the difference between the two sensor readings below which the end point is reached.	
		The threshold must be greater than the offset value from the vacuum test. See Chapter 7.4.3 "Determining offset value", page 52	
[Term]	Enter value	Specifies the length of time for which the pressure difference test is to be carried out.	

Precondition:

- ☑ A vacuum test has been carried out with a pressure equal to the pressure during the subsequent freeze-drying process. See Chapter 8.14 "Performing a vacuum test", page 78
- ☑ The sensors for the pressure difference test have been fitted. See "Connecting the sensors for a pressure difference test"
- ☑ The offset value has been determined. See Chapter 7.4.3 "Determining offset value", page 52
- \boxdot All flasks have been attached to the manifold drying rack.
- ▶ Navigate to [End point definition] via the navigation path.
- ► Tap [Pressure difference test].
- ► Set up the pressure difference test as desired.
- ► Tap [Start] to start the pressure difference test.

7.4.3 Determining offset value

Navigation path

 \rightarrow [Configuration] \rightarrow [End point determination] \rightarrow [Pressure difference test]

- Navigate to the [Pressure difference test] via the navigation path.
- \Rightarrow The control unit shows the offset figure.

7.4.4 Performing a temperature difference test

NOTE

The temperature test is successfully completed if all drying shelves are below the threshold.

The samples on a drying shelf have different drying times. Take account of the different drying times in the *[Duration]* setting.

The temperature difference test establishes the difference between the readings from the temperature sensor for the heatable shelf and the temperature sensor in the sample. If the difference between the two sensor readings is below a threshold, the freeze-drying phase can be ended.

Navigation path

→ [Method]

Precondition:

☑ The heatable shelves are fitted in the rack. See Chapter 7.9 "Operating top-mount drying racks", page 57

- ☑ The optional temperature sensor has been installed. See Chapter 7.9 "Operating top-mount drying racks", page 57
- ▶ Place the optional temperature sensor in the sample.
- ▶ Navigate to the *[Method]* menu via the navigation path.
- ▶ Tap the name of the method that you wish to edit.
- \Rightarrow The control panel highlights the selected method in green.
- ► Tap the action [Phase].
- \Rightarrow The control panel shows the action Phase.
- ► Tap the setting [End point definition].
- \Rightarrow The control panel shows the setting End point definition.
- ► Tap [Temperature difference test].
- \Rightarrow The control panel shows the temperature difference test.

The following settings are available:

Setting	Option	Explanation
[Temperature difference test]	Yes/No	Switches the temperature differ- ence test on or off.
[Start time]	Enter value	Sets the time from which the tem- perature difference test is to be performed. The value relates to the time before completion of the primary drying phase.
[Temperature difference limit]	Enter value	Specifies the difference between the two sensor readings below which the end point is reached.
[Term]	Enter value	The period of time during which the difference is not to be ex- ceeded. If the threshold is not ex- ceeded for the full period of time, the temperature difference test is passed.
[Continue]	Yes/No	Yes : the method switches to the next phase.
		No: the phase is ended when the set levels are reached.
[Message]	Yes/No	The control panel shows or does not show a message as soon as the temperature difference test is passed.

7.5 Performing freeze-drying using a method (Pro unit only)

7.5.1 Preparing the instrument

Time required:

approx. 30min

NOTE

i

To minimize the condensation of moisture from the air in the condenser, place a drying rack.

Navigation path

→ [Start]

- ► Navigate to the *[Start]* menu via the navigation path.
- ▶ Tap the function [Start conditioning] on the function bar.
- ⇒ The temperature in the ice condenser decreases to operating temperature.
- \Rightarrow The vacuum pump is brought up to operating temperature.
- After completion of the conditioning phase, the status bar shows the status Unload / Load.

7.5.2 Selecting a method

Navigation path

→ [Method]

- ▶ Navigate to the *[Method]* menu via the navigation path.
- ► Tap the function *[Method]* on the function bar.
- ► Tap the method that you wish to use.
- ▶ Tap the function [Activate] on the function bar.
- \Rightarrow The status bar shows the method activated.

7.5.3 Starting freeze-drying



Risk of skin burns from touching parts of the ice condenser after completion of conditioning.

Wear protective gloves when working on the instrument after the conditioning phase.

1	

NOTE

The freeze-drying process can be cancelled by tapping the functions [Manual] and [Aerate] on the [Start] menu.

Navigation path

→ [Start]

If a gas is being used

Precondition:

 $\ensuremath{\boxdot}$ The instrument has been prepared.

- \boxdot A method is selected.
- ► Fit a top-mount drying rack. See Chapter 7.9 "Operating top-mount drying racks", page 57.
- ► Load the top-mount drying rack with frozen samples.
- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function *[Start]* on the function bar.
- ▶ Make sure that the specified gas is being used.
- ► Answer **YES** to the confirmation question.
- \Rightarrow The freeze-drying process starts.
- ⇒ The control panel shows the *Start* menu with a black background.
- \Rightarrow The system carries out the selected method.

If a gas is not being used

Precondition:

 \square The instrument has been prepared.

- \square A method is selected.
- Fit a top-mount drying rack. See Chapter 7.9 "Operating top-mount drying racks", page 57.
- ► Load the top-mount drying rack with frozen samples.
- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function *[Start]* on the function bar.
- \Rightarrow The freeze-drying process starts.
- \Rightarrow The control panel shows the *Start* menu with a black background.
- \Rightarrow The system carries out the selected method.

7.5.4 Changing parameters while the process is running

• NOTE Comin

Coming step can be deleted.

- ▶ Select the step you wish to delete.
- ► Tap the [Delete] button on the function bar.

7.5.5 Ending freeze-drying

Navigation path

→ [Start]

Precondition:

 $\ensuremath{\boxdot}$ The status bar is showing the status **Hold**.

- ▶ Navigate to the [Start] menu via the navigation path.
- ▶ Tap the function [Aerate] on the function bar.
- ► Answer **YES** to the confirmation question.
- \Rightarrow The system is vented.
- \Rightarrow The status bar shows the status **Aerating**.
- ► Wait until the status bar shows the status **Unload / Load**.
- ▶ Remove the dried preparation from the top-mount drying rack.

NOTE

1

Shutting down after ending freeze-drying

For instructions on how to shut down the instrument after freeze drying, see Chapter 7.6 "Shutting down the instrument", page 55.

7.6 Shutting down the instrument

Time required:

50 min



NOTICE

Do not use force to remove the ice from the ice condenser.

Navigation path

→ [Start]

Precondition:

- $\ensuremath{\boxdot}$ The freeze-drying process has ended.
- ☑ No top mount drying rack is installed on the ice condenser.
- \square A container for draining is prepared.
- ☑ For instruments with ultimate vacuum control unit only: The vacuum pump is switched off.
- Navigate to the [Start] menu via the navigation path.
- ► Tap the function [Shut down] on the function bar.
- \Rightarrow The instrument is shutting down.
- After the instrument has shut down, the status bar shows the remaining time and the status Shutting down.
- ► Wait until the ice is completely thawed.
- Place a container with sufficient capacity underneath the drain valve.
- Turn the adjusting knob counter clockwise to open the drain valve manually.
- ⇒ The liquids drain into the container
- ► Wait and make sure that the condensate drain tube is drained completely.
- Clean the ice condenser. See Chapter 8.11 "Cleaning the instrument", page 77.
- Turn the adjusting knob counterclockwise to close the drain valve manually.



7.7 Defrosting the ice condenser coil by hot gas (optional)



Health risk due to exposal to vapor or liquid solvents.

- ► Keep an eye on defrosting progress inside the ice condenser chamber at any time.
- Avoid exposal to vapor.
- Avoid contact with the ice condenser materials containing liquid solvents at the end of the defrosting.

All operations and procedures in connection with the defrosting functionality of the instrument other than described in this chapter are not allowed and to be understood as misuse of the instrument.

Precondition:

- A fume hood or a sufficient air exhauster must be available to remove escaping vapors.
- ☑ Long defrosting periods must be avoided. Especially when aggressive solvents or acids are in the ice composition as these can lead to lifetime limitations because of corrosion or other material defects.

- ☑ The material stability of the drying chamber, seals, adapter plate and ice condenser exposed to liquid and vapor of defrosted solvents must be checked thoroughly to prevent long-term damage.
- ☑ Personal protection measurements like gloves, glasses and coat must be used while handling.
- \boxdot The instrument is in standby mode.
- ► Remove the drying chamber.
- ▶ Place the drain hose in a solvent/acid resistant tank.
- Press defrosting icon to initiate the defrosting for 60 min.
- ► Visually check the ice quantity inside the condenser.
- ► Select feasible duration to avoid vapor development during defrosting.
- Pre-long the defrosting time by additional 30 min by pressing the [+30 MIN] button.
- \Rightarrow The maximum extension is 8 x 30 min = 240 min (total of 300 min).
- ▶ Reduce the defrosting time by 30 min by pressing the [-30 MIN] button.
- If corrosive liquids are used in the ice condenser, open the drain valve during defrosting to release defrost water continuously.
- ▶ Interrupt the process at any time with the [STOP] button.
- Open the drain valve to release defrost liquid entirely.
- ▶ Wipe down the ice condenser and instrument to remove residual moisture.

7.8 Switching off the instrument

Precondition:

- ☑ The instrument has been shut down. See Chapter 7.6 "Shutting down the instrument", page 55.
- ▶ Switch the On/Off master switch to Off.

7.9 Operating top-mount drying racks

7.9.1 Operating stoppering acrylic drying chamber (heatable shelves)

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- ▶ Place the 300 mm dia. O-ring in the groove above the ice condenser.



condenser. ► Place the intermediate plate on the ice condenser. ► Loosen all fixing screws. ► Align the slots for the shelves. ► Tighten the fixing screws.

► Locate the springs in the holes on the ice



- ► Check the O-ring for damage.
- Screw the hand wheel upwards until the plate inside the lid has pressed the hooks all the way in.
- ▶ Place the O-ring into the groove of the cover.
- ▶ Place the cover on the cylinder.

- Make sure that the hooks on the rack are engaged.



► Carry out freeze-drying.

Precondition:

- $\ensuremath{\boxdot}$ The status bar shows the status **Stoppering**.
- Turn the hand wheel, until all samples are sealed.
- Confirm the verification question on the control panel.



7.9.2 Operating stoppering acrylic drying chamber (non-heatable shelves)

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- Place the 300 mm dia. O-ring in the groove above the ice condenser.



Locate the springs in the holes on the ice condenser.



Place the intermediate plate on the ice condenser.

► Loosen all fixing screws.







- ▶ Place the rack on the intermediate plate. ► Slide the shelves into the rack. Locate the cylinder in the groove above the main plate. ► Check the O-ring for damage. ► Screw the hand wheel upwards until the plate inside the lid has pressed the hooks all the way in.
- ▶ Place the O-ring into the groove of the cover.
- ► Place the cover on the cylinder.



Make sure that the hooks on the rack are engaged.



► Carry out freeze-drying.

Precondition:

 $\ensuremath{\boxdot}$ The status bar shows the status $\ensuremath{\textbf{Stoppering}}.$

- Turn the hand wheel, until all samples are sealed.
- Confirm the verification question on the control panel.



7.9.3 Determining the number of flasks for manifold drying chamber and rack



NOTE

Do not overfill flasks.

Maximum volume of the samples \leq half of the flask volume.



NOTE

Determine the number of flasks used individually for every process.



NOTE

The total volume of removing solvents should not exceed the ice capacity.

Maintain the pressure for a proper sublimation.

The number of flasks used is dependent on the following factors:

- The volume of the flask
- The method of freezing
- The concentration of the sample
- The composition of the solvent

Number of flasks using water as an example:

	Flask volume 1000 mL	Flask volume 500 mL
Rotating freezing	Max. 12 flasks	Max. 12 flasks

7.9.4 Operating manifold acrylic drying chamber (heatable shelves)

- ► Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- ▶ Place the 300 mm dia. O-ring in the groove above the ice condenser.



▶ Place the intermediate plate on the ice condenser.





► Align the slots for the shelves.









 Locate the cylinder in the groove above the main plate.



 Fit manifold valve onto the connection on the top-mount drying rack.



NOTE

Optionally, add a filter paper set (order no. 11067334, 11066144) to the manifold adapter.

For certain applications, the use of a filter paper set is recommended to prevent cross-contamination or blockage.

- ► Check the O-ring for damage.
- Place the O-ring into the groove of the cover.
- Place the manifold cover on the cylinder.



Load samples. See Chapter 7.9.3 "Determining the number of flasks for manifold drying chamber and rack", page 63.



► Carry out freeze-drying.

7.9.5 Operating manifold acrylic drying chamber (non-heatable shelf)

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- Place the 300 mm dia. O-ring in the groove above the ice condenser.
- Place the intermediate plate on the ice condenser.



▶ Place the rack on the intermediate plate.



 Locate the cylinder in the groove above the main plate.



 Fit manifold valve onto the connection on the top-mount drying rack.

- ► Check the O-ring for damage.
- ▶ Place the O-ring into the groove of the cover.
- ▶ Place the manifold cover on the cylinder.





NOTE

Optionally, add a filter paper set (order no. 11067334, 11066144) to the manifold adapter.

For certain applications, the use of a filter paper set is recommended to prevent cross-contamination or blockage.

Load samples. See Chapter 7.9.3 "Determining the number of flasks for manifold drying chamber and rack", page 63.



► Carry out freeze-drying.

7.9.6 Operating acrylic drying chamber (heatable shelf)

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- Place the 300 mm dia. O-ring in the groove above the ice condenser.



Place the intermediate plate on the ice condenser.



► Loosen all fixing screws.





(option)

- Turn the temperature sensor electrical connector so that the marks on the connector and the heatable shelf are parallel
- Press the temperature sensor electrical connector onto the connection.
- Locate the cylinder in the groove above the main plate.



- ► Check the O-ring for damage.
- ▶ Place the O-ring into the groove of the cover.
- ▶ Place the cover on the cylinder.



► Carry out freeze-drying.

7.9.7 Operating acrylic drying chamber (non-heatable shelf)

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- Place the 300 mm dia. O-ring in the groove above the ice condenser.







- ▶ Place the O-ring into the groove of the cover.
- ▶ Place the cover on the cylinder.



► Carry out freeze-drying.
7.9.8 Operating manifold drying rack

- Make sure that the groove above the ice condenser is clean, free of dust and not scratched.
- ► Check the 300 mm dia. O-ring for damage.
- Place the 300 mm dia. O-ring in the groove above the ice condenser.
- Fit manifold valve onto the connection on the top-mount drying rack.







NOTE

Optionally, add a filter paper set (order no. 11067334, 11066144) to the manifold adapter.

For certain applications, the use of a filter paper set is recommended to prevent cross-contamination or blockage.

Place the base plate on the ice condenser.





► Carry out freeze-drying.

7.9.9 Operating manifold valves

The lever on a manifold valve can be set to the following positions:



Position	Function
A: Lever pointing up	The connected vessel is evacuated.
B: Lever pointing down	The connected vessel is vented.

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 Maintenance work

Action		Daily	Weekly	Monthly	کر عبر Additional information
8.2	Cleaning the 300 mm O- rings	1			
8.3	Cleaning the top-mount drying racks	1			
8.9	Checking and replacing glassware components	1			
8.4	Cleaning the housing		1		
8.5	Cleaning and servicing the warning and directive symbols		1		
8.6	Cleaning the drain valve rotary knob		1		
8.7	Cleaning the ventilation slots			1	
8.8	Cleaning the drain valve membrane			1	
8.11	Cleaning the instrument			1	
8.12	Cleaning the vacuum tube				1
8.13	Cleaning the KF seals				1

1 - Operator

8.2 Cleaning the 300 mm O-rings

- ▶ Wipe down the 300 mm O-rings with a damp cloth.
- Check for scratches and other damage.
- ► If damaged, replace the 300 mm O-rings.

8.3 Cleaning the top-mount drying racks

- ▶ Wipe down the top-mount drying racks with a damp cloth.
- ▶ If heavily soiled, use a mild detergent.

8.4 Cleaning the housing

• Wipe down the housing with a damp cloth.

- ▶ If heavily soiled, use ethanol or a mild detergent.
- ▶ Wipe down the display with a damp cloth.

8.5 Cleaning and servicing the warning and directive symbols

- Check that the warning symbols on the instrument are legible.
- ▶ If they are dirty, clean them with a damp cloth.

8.6 Cleaning the drain valve rotary knob

► Turn the rotary knob clockwise until it can be removed from the instrument.



- ► Clean the rotary knob.
- ► Check the O-ring.
- ▶ Attach the rotary knob back on the instrument.

For applications containing alcohols and aggressive chemicals FFKM o-ring, spare part number 041639, should be used.

8.7 Cleaning the ventilation slots

Remove dust and foreign objects from the ventilation slots using compressed air or a vacuum cleaner.

8.8 Cleaning the drain valve membrane

- ▶ Remove the drain valve from the instrument.
- ▶ Clean the drain valve with a mild detergent.
- ▶ Check the sealings for damages.
- ▶ If any damages are visible, replace the sealings.
- Attach the drain valve back on the instrument.

8.9 Checking and replacing glassware components

- ► Check glassware components for damages before each use.
- ▶ Do not use glassware components that show any signs of damage.
- ▶ Immediately replace glassware components that show any signs of damage.
- ▶ Periodically replace glassware components.

8.10 Servicing the vacuum pump

 Carry out the maintenance according to the pump manufacturer's instructions. See relevant documentation.

8.11 Cleaning the instrument

- ► Do not spray or pour liquid on the instrument.
- ▶ Wipe down the instrument with a damp cloth.

8.12 Cleaning the vacuum tube

- Wipe down the vacuum tube with a damp cloth.
- Check the vacuum tube for scratches and other damage.

▶ If the vacuum tube is damaged, replace it.

8.13 Cleaning the KF seals

The following KF seals are located on the instrument:

- Seals ISO-KF 16
- Seals ISO-KF 25
- ▶ Wipe down the KF seals with a damp cloth.
- Check the KF seals for scratches and other damage.
- ▶ If the KF seals are damaged, replace them.

8.14 Performing a vacuum test



This procedure can only be performed with the advanced vacuum control unit.

The vacuum test checks the performance capacity of the vacuum system.

Time required:

NOTE

max. 10 min

Navigation path

→	[Configuration]	→	[Servicing] →	[Vacuum test]
---	-----------------	---	---------------	---------------

Precondition:

- \boxdot Conditioning of the instrument has been completed.
- \square A top-mount drying rack is fitted.
- ☑ The top-mount drying rack does not contain sample.
- ► Navigate to the action [Vacuum Test] via the navigation path.
- ► Specify a required setting for the vacuum to be achieved.
- Specify a required time within which the vacuum is to be reached.
- ► Tap the function [Start] on the function bar.
- \Rightarrow The vacuum test starts.
- \Rightarrow The status bar shows the status **Vacuum Test**.
- ⇒ If the vacuum pressure is not below 500 mbar after 30 s, the vacuum test automatically aborts.
- After completion of the vacuum test, the [Vacuum test] line shows whether the vacuum test has been passed or not.

Troubleshooting after failed vacuum test

Possible cause	Action
Top-mount drying rack not correctly fitted	Fit the top-mount drying rack correctly.
Top-mount drying rack is damaged	Check function of PMMA parts, replace manifold valves, clean drain valve.
O-rings dirty	Wipe down the O-rings with a damp cloth.
O-rings damaged	Check O-rings and replace if necessary.
Groove of O-rings dirty	Wipe groove of O-rings with a damp cloth.
KF clamps not closed	Close the KF clamps.
KF seals dirty	Wipe down the KF seals with a damp cloth.
KF seals damaged	Inspect KF seals and replace as neces- sary.

Possible cause

Action

The pump connected is not delivering suf- Carry out vacuum test with a different ficient performance vacuum pump.

8.15 Performing a leak test



NOTE

This procedure can only be performed with the advanced vacuum control unit.

8.15.1 Performing a leak test with a drying chamber

The leak test checks the vacuum system for possible leaks.

Time required: 45 min



NOTE

The leak test can be aborted by tapping [Stop] on the function bar.

Navigation path

→ [Configuration] → [Servicing] → [Leak test]

Precondition:

- \boxdot Conditioning of the instrument has been completed.
- \square A top-mount drying rack is fitted.
- \square The top-mount drying rack does not contain sample.
- ▶ Navigate to the action [Leak test] via the navigation path.
- On the [Ice Condenser] line, enter a required setting for the vacuum.
- ▶ On the [Shelf temperature] line enter the required shelf temperature.
- ▶ On the [Test scope] line select the component to be tested.
- ▶ On the [Drying shelf heating] line switch the drying shelf heating on or off.
- ▶ On the [Volume] line enter the actual volume of the components to be tested.

The volume of the system is calculated from the volume of the ice condenser and the top-mount drying rack.



36.46L

Acrylic drying chamber Acrylic drying chamber

43.41L

(with 4 drying shelves) (with 6 drying shelves)

- ► Tap the function *[Start]* on the function bar.
- \Rightarrow The leak test starts.
- \Rightarrow The status bar shows the status **Leak Test**.
- After completion of the leak test, the [Leak test] line shows whether the leak test has been passed or not.

⇒ The leak test is passed if the measured leakage rate is less than the pre-set rate of 10.10 mbar*L/h.

Troubleshooting after failed leak test

Possible cause	Action		
Top-mount drying rack not correctly fitted	Fit the top-mount drying rack correctly.		
O-rings dirty	Wipe down the O-ring with a damp cloth.		
O-rings damaged	Check O-rings and replace if necessary.		
Groove of O-rings dirty	Wipe groove of O-rings with a damp cloth.		
KF clamps not closed	Close the KF clamps.		
KF seals dirty	Wipe down the KF seals with a damp cloth.		
KF seals damaged	Inspect KF seals and replace as neces- sary.		
Pump oil dirty	Service according to manufacturer's in- structions.		
Leaking manifold valves	Replace leaking manifold valve.		
(manifold acrylic drying chamber only)	Locate leaking manifold valve. See Chap- ter 9.3 "Locating leaking manifold valve", page 85		

8.15.2 Performing leak test with a manifold drying rack

The leak test checks the vacuum system for possible leaks.

Time required: 45 min



NOTE

The leak test can be aborted by tapping [Stop] on the function bar.

Navigation path

→ [Configuration] → [Servicing] → [Leak test]

Precondition:

 \boxdot Conditioning of the instrument has been completed.

- ► Fit the base plate with a KF 40 blind flange, an ISO-KF 40 seal and an ISO-KF 40 clamp.
- ▶ Navigate to the action [Leak test] via the navigation path.
- ▶ On the *[Ice Condenser]* line, enter a required setting for the vacuum.
- ► On the [Shelf temperature] line enter the required shelf temperature.
- ▶ On the *[Test scope]* line select the component to be tested.
- ► On the [Drying shelf heating] line switch the drying shelf heating on or off.
- ▶ On the [Volume] line enter the actual volume of the components to be tested.

The volume of the system is calculated from the volume of the ice condenser and the top-mount drying rack.



13.64L

- ► Tap the function *[Start]* on the function bar.
- \Rightarrow The leak test starts.
- \Rightarrow The status bar shows the status **Leak Test**.
- ⇒ When the leak test is completed, the [Leak test] tab indicates whether the leak test was successful.
- ⇒ The leak test is passed if the measured leakage rate is less than the pre-set rate of 10.10 mbar*L/h.



NOTE

To test the leak-tightness of the manifold drying rack, carry out another leak test with the manifold drying rack installed.

Troubleshooting after failed leak test

Possible cause	Action		
Top-mount drying rack not correctly fitted	Fit the top-mount drying rack correctly.		
O-rings dirty	Wipe down the O-ring with a damp cloth.		
O-rings damaged	Check O-rings and replace if necessary.		
Groove of O-rings dirty	Wipe groove of O-rings with a damp cloth.		
KF clamps not closed	Close the KF clamps.		
KF seals dirty	Wipe down the KF seals with a damp cloth.		
KF seals damaged	Inspect KF seals and replace as neces- sary.		
Pump oil dirty	Service according to manufacturer's in- structions.		
Leaking manifold valves	Replace leaking manifold valve.		
(only if manifold drying rack installed)	Locate leaking manifold valve. See Chap- ter 9.3 "Locating leaking manifold valve", page 85		

9 Help with faults

9.1 Troubleshooting

Problem	Possible cause	Action		
Compressor fails to start or switches off	No power supply to compres- sor	Check the power supply and switch on the instrument.		
	Compressor overheated	 Allow the compressor to cool down. Check ambient conditions. 		
	Circuit-breaker has tripped	► Reset circuit of instrument.		
Cooling temperature is not achieved	Wrong ambient conditions	Adjust ambient conditions. See Chapter 3.5 "Technical data", page 19.		
	Too much ice in the ice con- denser	Defrost the ice condenser.		
System shuts down automatically	Major fluctuations in line power	 Connect instrument to an Uninterrupted Power Supply line. 		
Instrument does not work	Instrument is not connected to the power supply	 Make sure that the power supply is connected and switched on. 		
Main valve or vac- uum pump switches over frequently	Vacuum system is leaking.	 If necessary, replace tubing and/or seals. 		
Main valve does not switch over	Main valve fitted wrong way round	 Fit main valve correct way round (arrow pointing towards vacuum pump). 		
	Main valve lead not plugged in	Make sure that the electrical lead of the main valve is plugged into the correct socket on the rear of the instrument.		
Vacuum is not achieved	Vacuum system is leaking	 Clean the drain valve. Check manifold valve. See Chapter 9.3 "Locating leaking manifold valve", page 85 Check pressure regulation valve and aeration valve. See Chapter 9.4 "Finding a leaking regulation valve or aeration valve", page 86 If necessary, replace tubing and/or seals. 		
	Vacuum pump is too weak	 Use a vacuum pump with a capacity of at least 5m³/h. Carry out maintenance work according to the vacuum pump manufacturer's documentation. 		

Problem	Possible cause	Action
Instrument is not vented	Main valve is incorrectly con- nected	 Connect the main valve properly (see Chapter 5.4.2 "Fitting the advanced vacuum control unit", page 27).

9.2 Error messages

Error message	Possible cause	Solution
6040	Shelf set temperature is too high.	 Choose lower shelf set temperature.
6041	Shelf set temperature is too high.	Resolve any leakages.Choose lower shelf set
	Leakages.	temperature.
6042	Power failure has occurred.	 Recovery of the latest process parameters is started automati- cally. ▶ Check samples. ▶ Consider using a UPS.
6170	High sample load.	► Remove samples.
	High ambient temperature.	Check ambient temperature.Check fan.
6171	High sample load.	► Remove samples.
	High ambient temperature.	Check ambient temperature.Check fan.
6175	Separator inlet temperature sensor is disconnected.	Check cable connection.Measure sensor resistance.
	Defective component (e.g. sensor, cable, connector).	 Compare sensor resistance to PT-1000 table.
6176	Inlet temperature sensor on ice condenser is discon- nected.	 Check cable connection. Measure sensor resistance. Compare sensor resistance to
	Defective component (e.g. sensor, cable, connector).	PT-1000 table.
6177	Outlet temperature sensor on ice condenser is discon- nected.	 Check cable connection. Measure sensor resistance. Compare sensor resistance to
	Defective component (e.g. sensor, cable, connector).	PT-1000 table.
6179	Compressor outlet tempera- ture sensor is disconnected.	Check cable and connectors.Connect a functioning sensor.
	Defective component (e.g. sensor, cable, connector).	 Replace sensor.
6187	Stepper chip overtempera- ture.	Check cable and connector.Connect a functioning
	Undervoltage or short circuit at motor coil.	expansion valve.Replace the expansion valve.

Error message	Possible cause	Solution		
6188	Stepper chip overtempera- ture.	Check cable and connector.Connect a functioning		
	Undervoltage or short circuit at motor coil.	expansion valve.Replace the expansion valve.		
6189	Fan is not rotating.	► Check cable and connectors.		
	Fan is disconnected or blocked.	 Check if the fan rotates manually. 		
	Defective component (e.g. cable, connector).	Service.		
6190	High pressure sensor is dis- connected.	Check cable and connectors.Connect a functioning sensor.		
	Defective component (e.g. sensor, cable, connector).	 Replace sensor. 		
6191	Low pressure sensor is dis- connected.	Check cable and connectors.Connect a functioning sensor.		
	Defective component (e.g. sensor, cable, connector).	 Replace sensor. 		
6195	High sample load.	Remove samples		
	High ambient temperature.	 Check ambient temperature. 		
	Low air flow.			
6196	High sample load.	Remove samples		
	High ambient temperature.	Check ambient temperature.		
	Low air flow.			
6197	Compressor has no power.	Contact BUCHI Customer		
	Compressor is damaged.	Service.		
	Cable is broken.			
6198	Compressor error (voltage, motor or temperature error).	 Contact BUCHI Customer Service. 		
6272	Main valve is defective.	Check if the regulation valve		
	Regulation valve is defective.	can be opened.		
		aeration valve are swapped.		
		 Check if the valves are 		
		installed correctly.		
6273	Leaks after new flask attach-	► Stop flask attachment, until		
	closed or not tight.	pressure is below target		
		► Check if the vacuum regulation		
		valve is closed correctly.		
0074		Check for any leakages.		
6274	vacuum cannot be reached	 Resolve any leaks. Check oil condition 		
		 Check electrical connection of 		
		pump and fuses.		
6275	Pressure of 500 mbar cannot be reached within 30 s.	Resolve any leaks.		

Error message	Possible cause	Solution
6278	Pressure increases above level of 1.5 mbar during leak test.	 Resolve any leaks. Clean and dry ice condenser chamber.
	Leaks, moisture in the instru- ment.	► Clean O-rings.
6279	Leak test result is above al- lowed limit.	Resolve any leaks.Clean and dry ice condenser
	Leaks, moisture in the instru- ment.	chamber. ▶ Clean O-rings.
6280	Regulation valve is discon- nected.	 Check cable connection and solenoid condition.
	Defective component (e.g. cable, solenoid).	 Remove all vacuum valves to operate instrument with ultimate vacuum system.
6281	Aeration valve is discon- nected.	 Check cable connection and solenoid condition.
	Defective component (e.g. cable, solenoid).	Remove all vacuum valves to operate instrument with ultimate vacuum system.
6282	Main valve is disconnected.	Check cable connection and
	Defective component (e.g. cable, solenoid).	 solenoid condition. Remove all vacuum valves to operate instrument with ultimate vacuum system.
6283	Pressure sensor is discon- nected.	Check cable connection.Check condition of pressure
	Defective component (e.g. cable, sensor).	sensor.
6570	Heatable shelf is discon- nected.	 Connect a functioning shelf at the same connector position.
	Defective component (e.g. heating coil, cable, plug).	 Contact BUCHI Customer Service.
6571	Sample temperature sensor is disconnected.	 Connect a functioning PT-1000 at the same connector position
	Defective component (e.g. cable, plug).	 Contact BUCHI Customer Service.
6670	Stoppering is disconnected.	 Check cable connection. Contact BUCHI Customer Service.

9.3 Locating leaking manifold valve

Navigation path

→ [Start]

Precondition:

- \boxdot The system does not evacuate to less than 0.1 mbar.
- ► Navigate to the *Start* view via the navigation path.
- Set the vacuum to 0.5 mbar.
- ► Tap the function *[Start]* on the function bar.

- Turn each manifold valve individually while checking the display to see if the actual pressure changes.
- If the vacuum decreases in the case of one of the manifold valves, then that valve is the one that is leaking.
- ► Tap the function [Aerate] on the function bar.
- Replace the manifold valve concerned.

9.4 Finding a leaking regulation valve or aeration valve

Navigation path

→[Start]

Precondition:

 \boxdot The instrument has been prepared.

- ▶ Remove regulation or aeration valve.
- ► Seal the opening with KF16 cover.
- Fit a top-mount drying rack. See Chapter 7.9 "Operating top-mount drying racks", page 57.
- ▶ Navigate to the [Start] menu via the navigation path.
- ► Tap the function *[Manual]* on the function bar.
- Enter the specified settings for the vacuum.
- ► Tap the function [Start] on the function bar.
- \Rightarrow The background color of the *Start* menu changes from white to black.
- \Rightarrow The status bar shows a clock counting up and the status **Manual Drying**.
- \Rightarrow The system evacuates to the set pressure.
- \Rightarrow If the specified setting is not reached, the value is leaking.
- ► Carry out the instructions for action on the other valve.

10 Taking out of service and disposal

10.1 Taking out of service

- ▶ Switch off the instrument and disconnect it from the mains power supply.
- ▶ Remove all tubing and communication cables from the instrument.

10.2 Disposal

The operator is responsible for proper disposal.

- When disposing the instrument, equipment or packaging, observe the local regulations and statutory requirements regarding waste disposal.
- When disposing, observe the disposal regulations of the materials used. For the used materials see Chapter 3.5 "Technical data", page 19 or the material labeling on the parts.
- ► The customer is encouraged to recycle suitable materials.



NOTICE

Environmental hazard from flammable refrigerant

The refrigerant used to operate the instrument is flammable.

 Dispose the instrument properly, if necessary using a professional disposal service.

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.

11.1.1 Accessories

	Order no.	Image
Main valve	11076675	
Pressure regulation valve	11076679	
Aeration valve	11064724	
Pirani / Piezo pressure sensor	11062228	
Capacitive pressure sensor Inficon CDG 020 D	11062230	
PT1000 sample temperature sensor	11064031	
Set for pressure difference test incl. extension cable for PPG011 and CDG 020D sensors, PMMA top cover, seals and clamps	11067590	

	Order no.	Image
Set for pressure difference test incl. extension cable of PPG011 and CDG 020D sensors, stoppering top cover, seals and clamps	11070102	
Set for pressure difference test	11080770	
Only for manifold rack steel with 12 connections		
Trolley L-250, stainless steel coated	11080440	
SD-Card 1 GB	11064730	
Connection cable, for vacuum pump	11064934	
Vacuum pump Pfeiffer Duo 6	11065652	
Vacuum pump Edwards nXDS6iC	11068158	

11.1.2 Top-mount drying rack accessories

	Order no.	Image
Drying chamber tube, PMMA, for 4 shelves	11063278	
L 368 mm, Ø 300 mm		
		\bigcirc
Drying chamber tube, PMMA, for 6 shelves	11065093	\bigcirc
L 480 mm, Ø 300 mm		
		\bigcirc
Top cover, PMMA, without sealing	11062912	
Ø 300 mm, H 50 mm		

	Order no.	Image
Top cover manifold, PMMA, with 12 valves, without sealing Ø 300 mm, W 546 mm, H 127 mm	11065595	
Top cover stoppering, without sealing only for rack for 4 heatable shelves Ø 300 mm, H 330 mm, W 320 mm	11064314	
Rack for 4 heatable shelves H 356 mm, Ø 265 mm , shelf distance 30-75 mm	11065102	
Rack for 6 heatable shelves H 468 mm, Ø 265 mm, shelf distance 30-75 mm	11065103	
Heatable shelf, aluminum coated, with con- nection cable Ø 219.5 mm, surface area 376 cm ²	11064095	
Sample tray, stainless steel Ø 220 mm, H 18.5 mm	11061439	
Ferrule, Ø 218 mm, H 40 mm	11065816	
Drying rack manifold, stainless steel, with 12 valves H 340 mm, W 777 mm	11063664	
Drip pan, for manifold	11066358	

	Order no.	Image
Support for rack, stainless steel	11063789	
H 4 mm		
Baseplate, stainless steel, for manifold rack	11064953	
Baseplate, PMMA, for manifold rack	11065733	
Suction nipple	11065819	~
For manifold application to create vacuum in sample flasks		
Ampoule adapter for manifold	11065725	9
with 19 ampoule connections and cap adapter		
Manifold valve, EPDM/Silicone, with SJ 29/32	11062300	
Flask beaker for manifold 100 mL	11066140	
with cap adapter and integrated filter		
Flask beaker for manifold 200 mL	11066141	
with cap adapter and integrated filter		
Flask beaker for manifold 800 mL	11069474	(· 9 ·)
with cap adapter and integrated filter		

	Order no.	Image
Flask beaker for manifold 1200 mL	11066143	
with cap adapter and integrated filter		
Manifold flask adapter set	11066144	
with 12 adapters, incl. filter paper		\bigcirc
Manifold flask adapter set	11067334	\bigcirc
with 6 adapters, incl. filter paper		\bigcirc
		\bigcirc
		\bigcup
Manifold flask adapter set, 24/40 joint size	11066171	\frown
with 12 adapters, incl. filter paper		\bigcirc
		\bigcirc
		\bigcup
Manifold flask adapter set, 24/40 joint size	11067333	\bigcirc
with 6 adapters, incl. filter paper		\bigcirc
		\bigcirc
		\bigcup
Filter round 20mm Set à 100pcs	11065801	
Filter for manifold valve		
Filter round 47mm Set à 100pcs	11065731	
Beaker flasks with volume above 600 mL		
Filter round 30mm Set à 100pcs	11065728	
Beaker flasks with volume below 600 mL		
Unheatable shelf for stoppering	11079991	

11.1.3 Spare parts

	Order no.	Image
O-Ring Ø 300 mm	11065367	
Vacuum hose, KF 25, L 1000 mm	11066031	
Vacuum clamp KF 16	11064939	
Vacuum seal KF 16	11063455	
Vacuum seal KF 25	11063457	Ø
Vacuum seal KF 40	11063659	Ø
Vacuum flange adapter, stainless steel, KF 16 to KF 25	11064870	
Blind flange KF 16	11064902	

	Order no.	Image
Blind flange KF 25	11063660	
Blind flange KF 40	11063661	

11.1.4 Wear parts

	Order no.	Image
Mineral oil 1 L vacuum pump Pfeiffer DUO 6	11065664	
Tip seal kit for vacuum pump Edwards nXD- S6iC	11069617	
Seals for drain valve, FFKM	041639	

11.1.5 Software

	Order no.
Lyovapor™ software licence	11065668
Lyovapor™ software USB	11065667

11.1.6 IQ/OQ kits

	Order no.
IQ/OQ set en	11081555
Repeating OQ en	11081556

11.1.7 Maintenance kits

	Order no.
omer kit for L-250 / L-210, with advanced vacuum control	11076718
uently used wear and spare parts for self-servicing your device, eniently gathered in one kit	
omer kit for L-250 / L-210, with ultimate vacuum control	11076719
uently used wear and spare parts for self-servicing your device, eniently gathered in one kit	



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