

Frozen in Time Ltd

Manufacturers of Freeze Drying Machines and Vacuum Cold traps

Operating Manual

Lablyo Ultra

Main associated accessories:

Acrylic drying chamber	FIT/LYO/01/0
6 height shelf rack	FIT/LYO/03/0
6 heated shelf rack	FIT/LYO/19/0
4 shelf vial closing device	FIT/LYO/22/0
8 port flask manifold	FIT/LYO/13/0
48 port ampoule manifold	FIT/LYO/25/0
Spin Freezer	FIT/LYO/28/0
Vacuum pump for Lablyo Ultra	FIT/LYO/59/0

Includes IQ/OQ Qualification



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1. General Information

1.1. Introduction

What is freeze drying (lyophilisation)?

Freeze drying means: Removal of water from frozen material. The drying process takes place by direct conversion from ice to vapour. This process is called sublimation. Sublimation happens under vacuum when the temperature in the product is less than -10°C .

The aim of freeze drying is to obtain a readily water-soluble product which has the same characteristics as the original product after the addition of water. As the drying process takes place in the frozen state at low temperatures it is possible to dry proteins which will not denature. Most of the other chemical compounds will also remain unchanged.

Freeze drying products, of biological origin such as tissues, tissue extracts, bacteria, vaccines and sera transforms them into a dry product. During this process enzymatic, bacterial and chemical changes are largely avoided. Freeze drying is the gentlest process for preserving the biological properties of sensitive tissue and tissue components. Freeze drying can also be used for dry some inorganic products.

1.2. Applications

The **Lablyo Ultra** is a laboratory and pre-production machine for the freeze drying of products in ampoules, vials, glass flasks, bottles or dishes. The initial freezing of the products will require a spin freezer, flask freezing bath, laboratory freezer or in some cases the pre freezing of the products can be done in the **Lablyo Ultra** chamber. The appropriate accessories are required for freeze drying can be made with the use of the.

1.3. Technical data of freeze dryer

Lablyo Ultra

Performance data	-55°C
Ice condenser capacity	10 kg max.
Ice condenser performance ¹⁾ :	6 kg per 24 hour maximum
Physical data	
Dimensions of the unit:	width: 460 mm height: 930 mm depth: 630 mm
Weight:	approx. 48 kg
Noise emissions according to DIN 45635:	54 dB(A)
Chamber Dimensions	Ø270mm x 500mm
Electromagnetic compatibility according to EN 55011:	class B
Refrigerant:	R507 - 1.4 KG
Voltage	240V 50Hz (220V 60Hz special order)
Main fuse rating:	13 Amp
Ambient temperature	5°C to 30°C
Equipment connections:	
Vacuum connection:	19mm nozzle
Drain valve	1/4 BSP ball valve
Electrical connection	IEC connection

1.4. Safety instructions

1.4.1. Disconnect the mains plug before removing panels

The mains plug must be disconnected before the panels are removed or any maintenance work is undertaken..

1.4.2. Solvents

Acidic or high solvent concentration products should not be dried because of corrosion risk and damage to the vacuum pump.

1.4.3. Cleaning and Maintenance of the Unit

For infectious, toxic, pathogenic and radioactive substances, the danger information of the associated safety regulations must be observed.

1.4.4. Freezing of skin to surfaces

Make sure skin does not come into contact with freezing surfaces. Skin can only be detached from the surface by applying heat. Do not use liquid.

1.4.5. Transporting

The **Lablyo Ultra** should be carried by two people and care must be taken when placing the unit down on a surface not to trap fingers. .

1.5. The Lablyo Ultra should not be used when:

1. It is not properly installed.
2. The lid or accessories are not properly installed.
3. Panels are missing.
4. The operator is not authorized or trained
5. Highly corrosive or solvent substances are present.
6. The accessories are not designed to be used with the unit or show signs of damage. Glassware can be susceptible to implosion causing potential risk during freeze drying.
7. In hazardous or dangerous locations.
11. The products are explosive or highly flammable.
12. The products are Infectious, toxic, pathogenic or radioactive unless in suitable vessels and in accordance with the relevant safety data.

2. Information on Freeze Drying

2.1. General Information on Freeze Drying

Freeze drying is the gentlest process for drying products. It uses the process of sublimation, the direct conversion from solid to gaseous state. The frozen product is dried under vacuum without thawing. The condenser chamber works as a cryogenic pump as it takes large quantities of vapour and condenses it to ice. The vacuum pump is only intended to remove the air from the drying chamber but not the vapor. In order to start the sublimation process, energy must be supplied to the product.

When drying takes place in glass flasks on a manifold, the heat energy is taken from the warmer surrounding air.

When drying takes place on shelves in an acrylic chamber, the heat energy source is radiation from the environment and directly by from the shelves if they are heated.

Primary drying removes the most of the water from the product. Secondary drying removes the last traces of water means of deep vacuum..

The main components of a freeze dryer are:

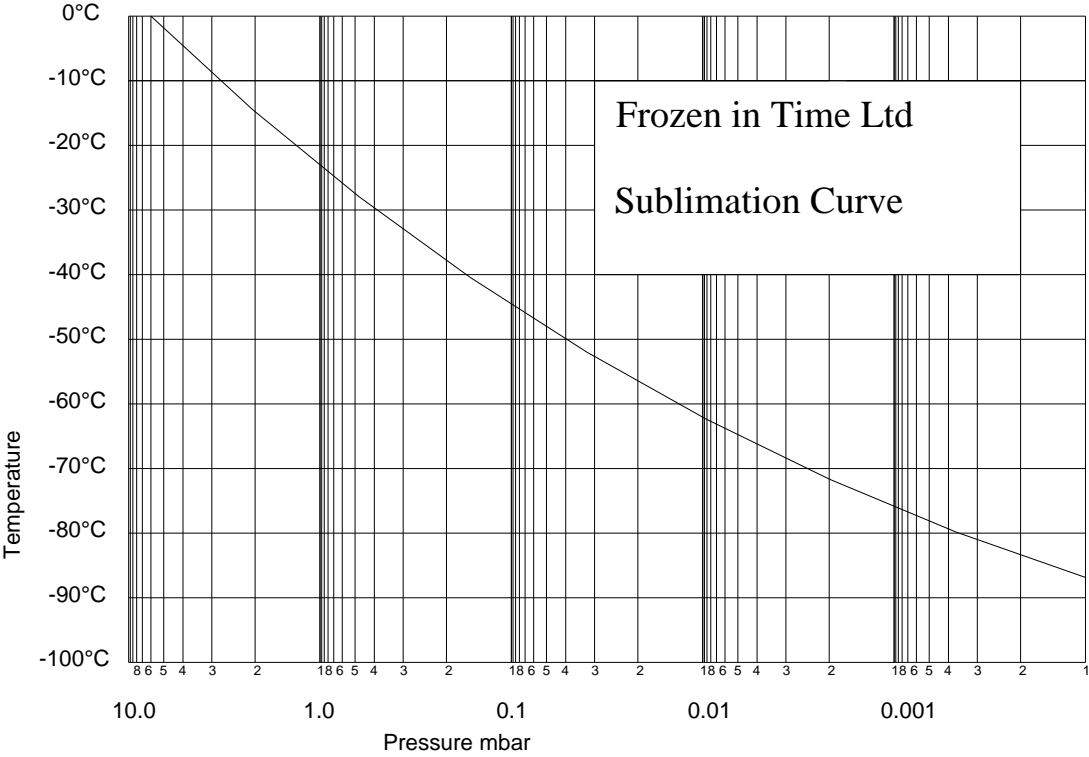
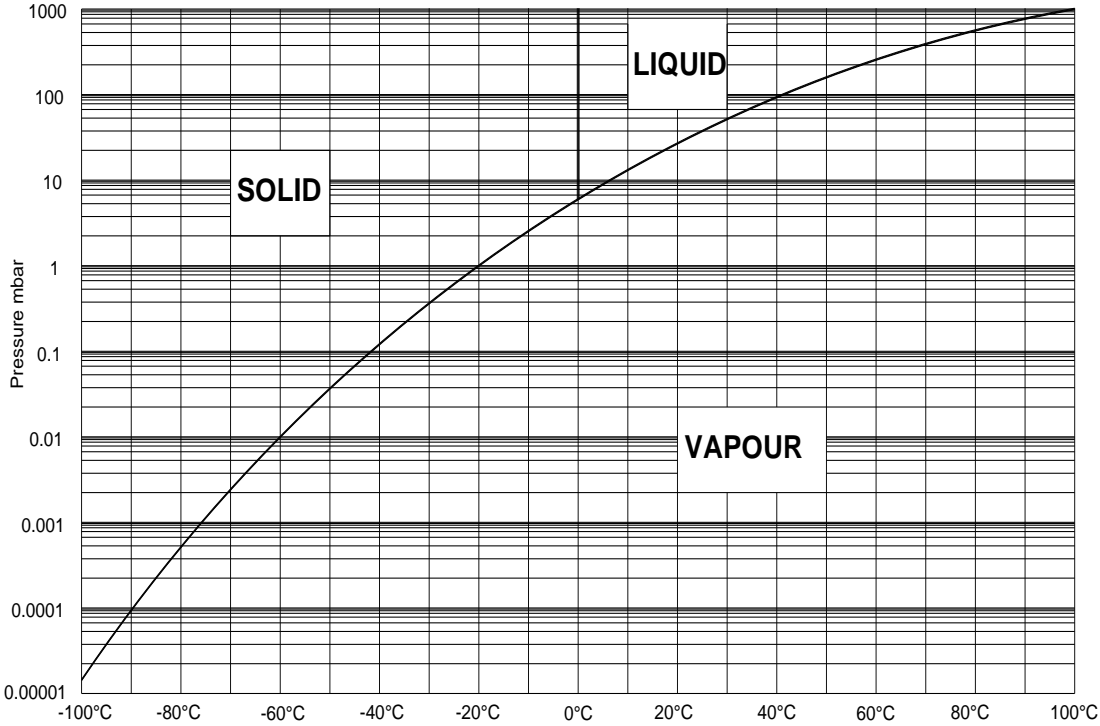
- Vacuum drying chamber or manifold
 - a) plain or heated shelves for drying in dishes
 - b) shelves with sealing device for drying in vials or small bottles
 - c) rubber valves for connecting round-bottom flasks, bottles, etc.
 - d) rubber nipples for connecting ampoules
- Vacuum pump to evacuate air.
- Ice condenser with a temperature of -55°C to condense vapour.

Sublimation

The principle of sublimation is briefly explained using the phase diagram of solid liquid and vapour. If the pressure is higher than 6.1 mbar, water passes through all three phases (solid, liquid, vapour) when the temperature is lowered or raised. At 6.1 mbar and 0°C all three lines meet, this is called the triple point where all three phase can occur simultaneously. Below this point when the pressure is lower than 6.1 mbar, the ice is converted directly from a solid to a vapour on reaching the sublimation pressure curve.

The sublimation curve shows the vapour pressure of water as affected by temperature and pressure.

Phase diagram for solid, liquid and vapour



2.2. Freezing

Small product quantities can be frozen directly inside the ice condenser chamber of the **Lablyo Ultra**. Larger quantities are pre-frozen in a deepfreeze. This the usual option for product to be freeze dried in trays or vials. Deep fill depths should be avoided by using wider containers filled to a shallower depth in order to maximize surface area. Liquids to be dried in narrow ampoules should be frozen in a spin freezer this causes the product to freeze in a thin section up the wall of the tube. Liquid to be dried in flasks should be rotated in a freezer bath. This causes the frozen material to form a thin layer, lining the flask. These procedures will help minimize the drying time.

It is advisable to pre-cool the shelves in order to avoid partial thawing during the evacuation.

Products containing solvent, high salt or sugar concentrations require freezing to lower temperatures.

2.3. Primary drying

The **Lablyo Ultra** is taken to its operating temperature.

The vacuum pump is switched on.

The duration of the main drying phase depends mainly on:

- the layer thickness of the product,
- the solid content of the product,
- the heat supplied to the product during the drying process,
- the vacuum pressure inside the drying chamber during the drying process.

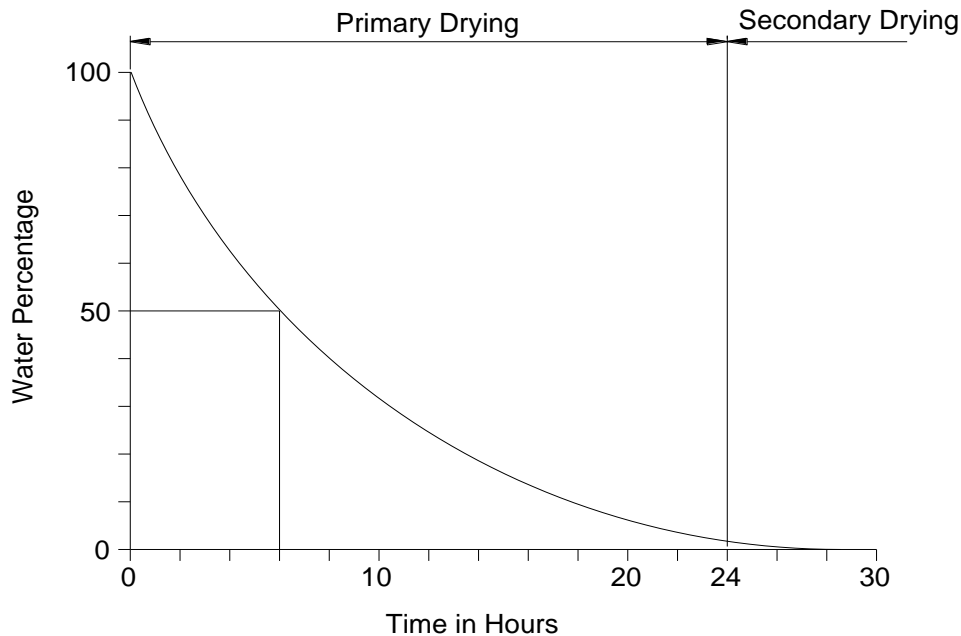
With increasing pressure the rate of sublimation rises as long as it stays below the vapour pressure of the product. This is because at higher pressures the heat energy reaches the sublimation front of the ice core sooner. Therefore the drying period is shortened. The water vapor generated during the main drying phase is not intended to be removed by the vacuum pump. It is to be collected by the ice condenser. The purpose of the vacuum pump is to lower the partial pressure of the non-condensable gases so that the water vapor can be transported from the product to the ice condenser. However, small quantities of water vapor will be removed by the vacuum pump. The vacuum pump is equipped with a gas ballast valve that when open, removes traces of condensable vapors from the pump. For this reason the gas ballast valve can be open during the main primary phase. The gas ballast valve is not required for secondary drying and closing it will help achieve a lower level of vacuum.

During primary drying the moisture is removed by sublimation and during final drying the bound moisture is removed by desorption. The recommended vacuum pump should reach with open gas ballast valve, a vacuum level lower than the relevant water vapour pressure.

The residual moisture of the dried product depends mainly on:

- The temperature of the dried product during the final drying process,
- The final vacuum reached during the final drying process.

The end of the primary drying phase is reached, when the product temperature is nearly the same as the shelf temperature (temperature difference between shelf and product approximately 3 to 5°C). Once the primary drying is completed the secondary drying will remove the bound water from the product. The following diagram shows the drying process for a product containing approximately 10 % solid matter. During the first quarter of the primary drying phase 50% of the water content is condensed. During the next quarter of the primary drying phase 50% of the remaining water content is condensed. This continues until the drying curve approaches the time axis asymptotically. This typical drying curve is due to the fact that the area of sublimation recedes into the product and the remaining water vapour must pass through the already dried layers. During the drying process the resistance increases. The drying curve is determined by the latent heat of sublimation and the amount of vapour transported. In order to increase the specific heat conduction properties of the product and to keep the water vapour volume as low as possible it is necessary that drying takes place as close as possible to the solidification point or eutectic point.



The drying time depends heavily on the vacuum level. The nearer the vacuum to the solidification point in accordance with the vapor pressure curve the shorter the drying time.

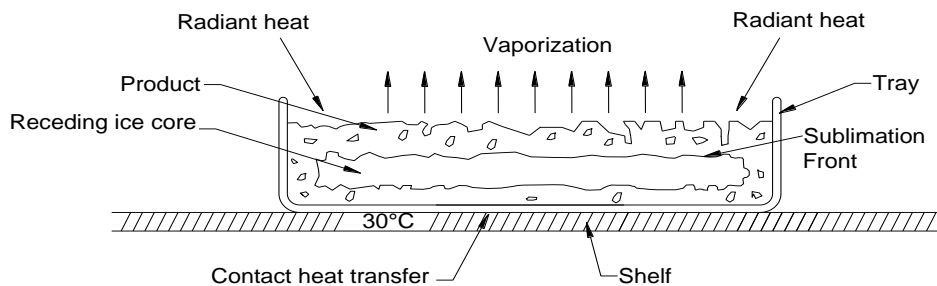
Facts regarding ice in a vacuum:

- 1.0 gram of ice at;
- 1.0 mbar assumes a volume of 1 m³ vapor
- 0.1 mbar assumes a volume of 10 m³ vapor
- 0.01 mbar assumes a volume of 100 m³ vapor

Heat supply during drying

The required heat supply to the product to be dried takes place by;
Conduction through contact in the drying chamber
Mild conduction through low pressure vapour
Radiant heat energy.

Affects of freeze drying of a product in a dish



Heat transfer takes place via the heated shelves by direct contact with the bottom of the tray. At the beginning of sublimation the transfer of heat is very effective from the wall of the tray to the frozen product. However, soon an area develops which is ice-free, porous, dried and has an insulating effect between the wall of the tray and the product. This slows down the heat energy transfer available to the ice core. The porous dried layer enables the passage of vapour from the ice core. If it is restricted the temperature will increase and ice core will thaw rather than sublime. This applies especially to inhomogeneous products and to great layer thicknesses. During this drying phase it is important to regulate the heat supply and control temperature and pressure precisely.

2.4. Secondary drying

The final pressure in the drying chamber depends on the ice condenser temperature according to the vapor pressure curve above ice :

- e. g. 1.030 mbar correspond to -20°C
- 0.370 mbar correspond to -30°C
- 0.120 mbar correspond to -40°C
- 0.040 mbar correspond to -50°C
- 0.01 mbar correspond to -60°C

The unit is in operating condition if the temperature of the ice condenser is lower than -50°C and the pressure is lower than 0.12 mbar. The final pressure measured when there is no product in the unit and its corresponding ice temperature is determined by the warmest ice surface in the condenser chamber.

2.5. End of drying and air admittance

The product is dry when it is at or above ambient temperature while under the secondary drying vacuum level. The condenser temperature will also be lower than when under load.

The vacuum pump can be switched off and the condenser chamber drain valve can be opened as an air admittance valve. This valve can be used to purge the unit with nitrogen or another inert gas instead of using air. Then the unit is switched off and the product is removed.

2.6. Defrosting

Defrosting of the ice condenser is carried out by switching off the refrigeration and switching on the red defrost button. To let the water out, open the drain valve and allow it to drain into an appropriate container. The defrost is self-regulating and will not overheat.

3. Description of the Freeze Drying Processes

3.1. Freezing the product.

There are 4 ways to freeze the product when using the Lablyo Ultra

1. The condenser chamber
2. A laboratory freezer preferably -40°C
3. The Spin freezer
4. Flask freezing in a glycol bath

3.1.1. Freezing inside the ice condenser chamber.

Possible water residue is removed from the ice condenser chamber. To do this open the drain valve so that water residue can drain off. Then the ice condenser chamber should be wiped out if necessary. This



will minimize the risk of trays freezing to the chamber. The refrigeration unit is then switched on and allowed to go down to operating temperature. The trays can then be lowered into the condenser chamber to freeze. With vials, several layers of trays can be stacked into the chamber at once. A blank lid can be placed on the top of the chamber to keep warm air and moisture from entering while the product is freezing. The same principle can be used for tray drying although more care is needed when stacking the trays.

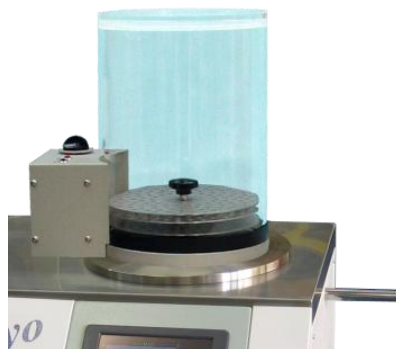
3.1.2. Freezing in an external freezer.

The product is prepared in vials or trays and then placed in a -40 °C freezer until it is completely frozen.

3.1.3 The Spin freezer

The spin freezer prevents foaming when vacuum freezing is used. It does this by using centrifugal force.

The long thin ampoules sit at an angle with the tops facing in slightly towards the centre. The vials are left to spin on an adjustable timer while the vacuum is applied. The centrifugal force will stop the product from overflowing and a long thin tapered layer will freeze up the side of the slanting ampoule maximizing the surface area.



3.1.4. Flask freezing in a glycol bath

The flasks should only be partly filled then rotated in the freezing glycol so a thin layer freezes evenly around the inside of the flask to maximize the surface area.

3.2.1. Drying on plain shelves

Vials, bottles, trays or dishes containing frozen product are arranged on the shelves and lowered into the acrylic chamber.

These shelves are ideal for product that requires slow drying with minimal heat input or small volume drying.



3.2.1. Drying on heated shelves

Vials, bottles, trays or dishes containing frozen product are arranged on the shelves and lowered into the acrylic chamber. These shelves are ideal for product that requires faster drying or higher temperatures. Each shelf is has equal heat input and there is a temperature sensor sampling one shelf only. Product should be loaded equally over all the shelves to allow for even drying rates. There are leadthroughs in the lid for the temperature sensor and power supply.

The **Lablyo Ultra** requires a separate temperature controller.

3.2.3. Drying in a closing device

The closing device is a series of shelves, also for use inside an acrylic chamber. Trays of frozen product are loaded onto it is then lowered into the chamber. When the product is dry the lids on the vials can be closed sealing in either a vacuum or a nitrogen charge. To do this the handle is turned clockwise to close levels 1 and 3 and then anticlockwise to close levels 2 and 4.



3.2.4. Drying in a closing device with heated shelves

This combines the closing device with the heated shelves in one unit. For use when extra heat is required in closable vials.

3.2.5. Freeze drying with a column or lid manifold

These manifolds use rubber valves to attach the glass freeze drying flasks. The flasks are frozen first and then can be added or removed from the manifold while the refrigeration and vacuum pump are running.

When adding a flask to a manifold that is under vacuum it is important to close the valves on the other flasks that are in use. Open the valve to the newly introduced flask and after a few minutes the rest of the valves can be reopened.



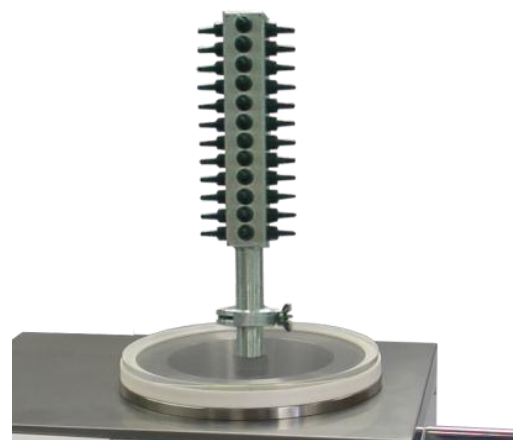
3.2.6. Freeze drying with a 48 port ampoule manifold

The 48 port ampoule manifold requires that all 48 ports are used at once.

Ampoules from the spin freeze are a push on seal to the rubber nipples. When the frozen the ampoules are attached then will freeze dry from the vacuum and the ambient heat.

Once the product is dry the ampoules can be removed and sealed.

The ampoules are usually hermetically sealed using a flame to melt the glass tube while it is being twisted.



3.2.7. The Lablyo Ultra as a standalone cold trap

The Lablyo Ultra can be equipped with an adaptor lid to act as a cold trap for vacuum pump protection. It will trap water vapour or any other condensable that would contaminate the vacuum pump oil. It can also be used for vacuum distillation so the condensable product can be collected.

4. Installation and Commissioning of the Unit

4.1. Site of installation

In order to ensure the air circulation of the heat exchanger, do not place any paper, cloths or similar items behind the unit. The freeze dryer should be positioned horizontally. The ambient temperature should be between approx. +5°C and +30°C. The refrigeration compressor of the freeze dryer is air-cooled. Sufficient air circulation must be ensured. A distance of at least 30cm to the wall should be kept. The unit should not be positioned near radiators or heat sources. In the case of insufficient air circulation or too high ambient temperatures, the temperature and the pressure in the refrigerating system will increase. If the maximum permissible operating pressure is exceeded, this may cause the refrigeration unit to switch off. The following connections are required at the site:

4.2. Mains power

The operating voltage on the name plate must correspond to the local supply voltage. Frozen in Time freeze dryers are units of safety class I. The **Lablyo Ultra** has a three-wire connection cable with a 230VAC 13 amp fused plug earth contact. The freeze dryer must be on a circuit protected with a 16 Amp fuse or circuit breaker.

4.3. Air admittance / Drain valve

The drain valve is also used for air admittance. If bottles have to be sealed in nitrogen the ice condenser chamber can be purged through the drain valve.

Take care not to over pressure the system.

4.4. Condensate and defrosting water

The condensate and the defrosting water are discharged through the drain valve.

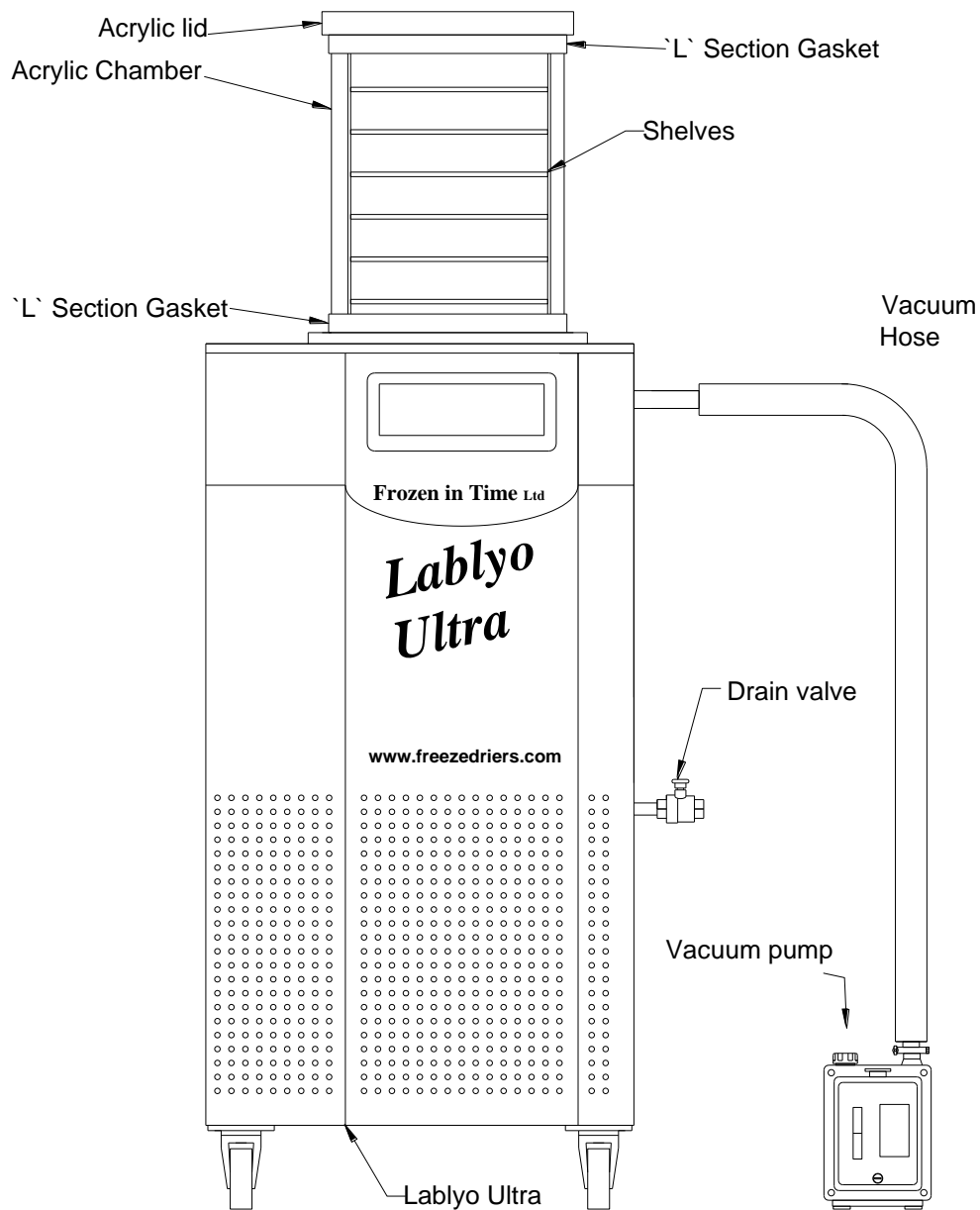
4.5. Vacuum pump exhaust gases

The oil mist from the vacuum pump is normally trapped in an exhaust filter.

If this is not the case, the oil mist has to be discharged. A hose can be connected to the exhaust flange of the vacuum that leads into the open air or a vent. During installation of the pipe special care must be taken that condensate cannot flow back into the pump.

4.6. Initial start-up

Prior to start-up, make sure that the freeze dryer has been properly set up and installed

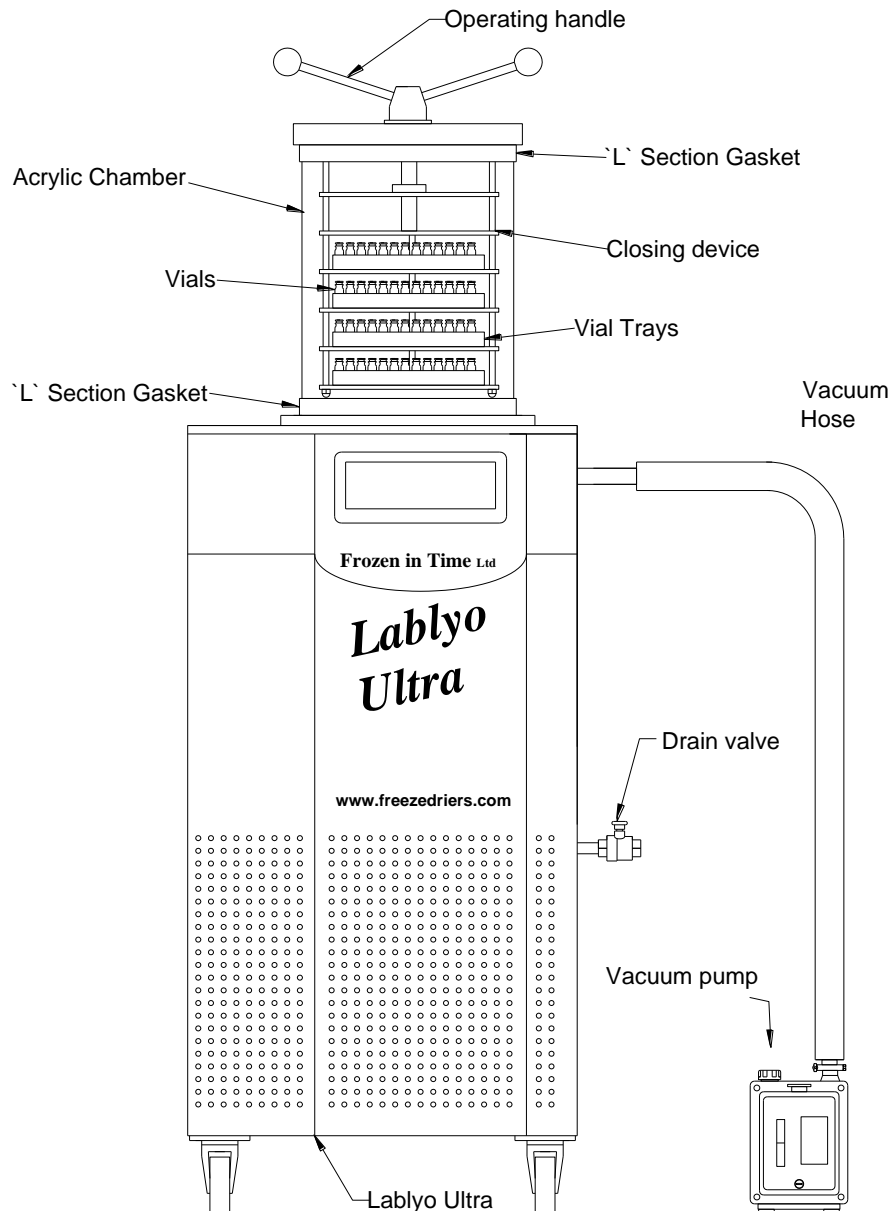


Installation of the vacuum pump

The vacuum pump can be powered through the Lablyo Ultra via the IEC socket on the left hand side. Make sure that the vacuum exhaust gases are filtered or carried off.

4.7. Installation of accessories

4.7.1. Vial closing device



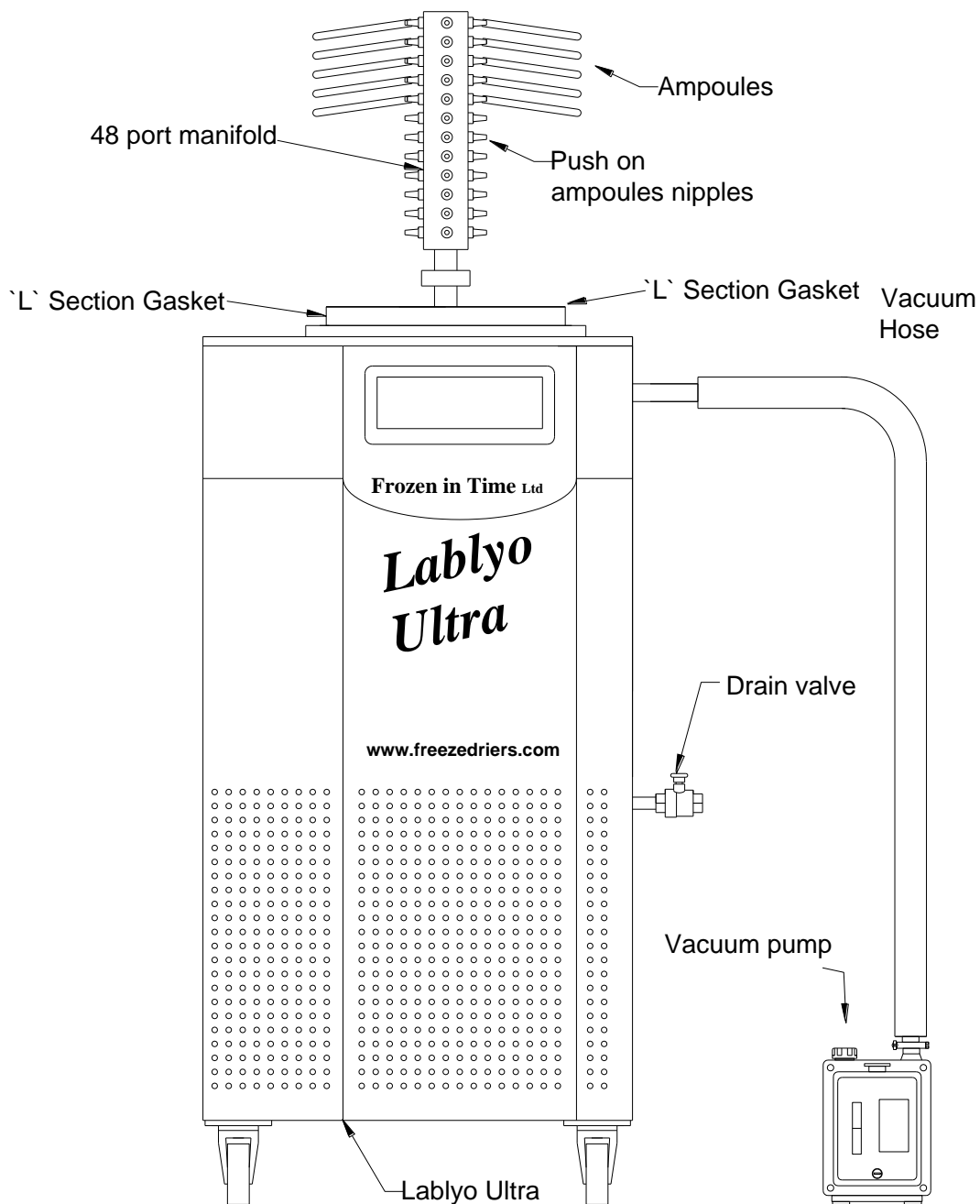
FIT/LYO/22/0

Vial Closing Device

Additionally requires:

Acrylic drying chamber	FIT/LYO/01/0
2 x `L` section gaskets	FIT/LYO/40/0
4 x Trays 230mm	FIT/LYO/34/0

4.7.2. 48 port ampoule manifold



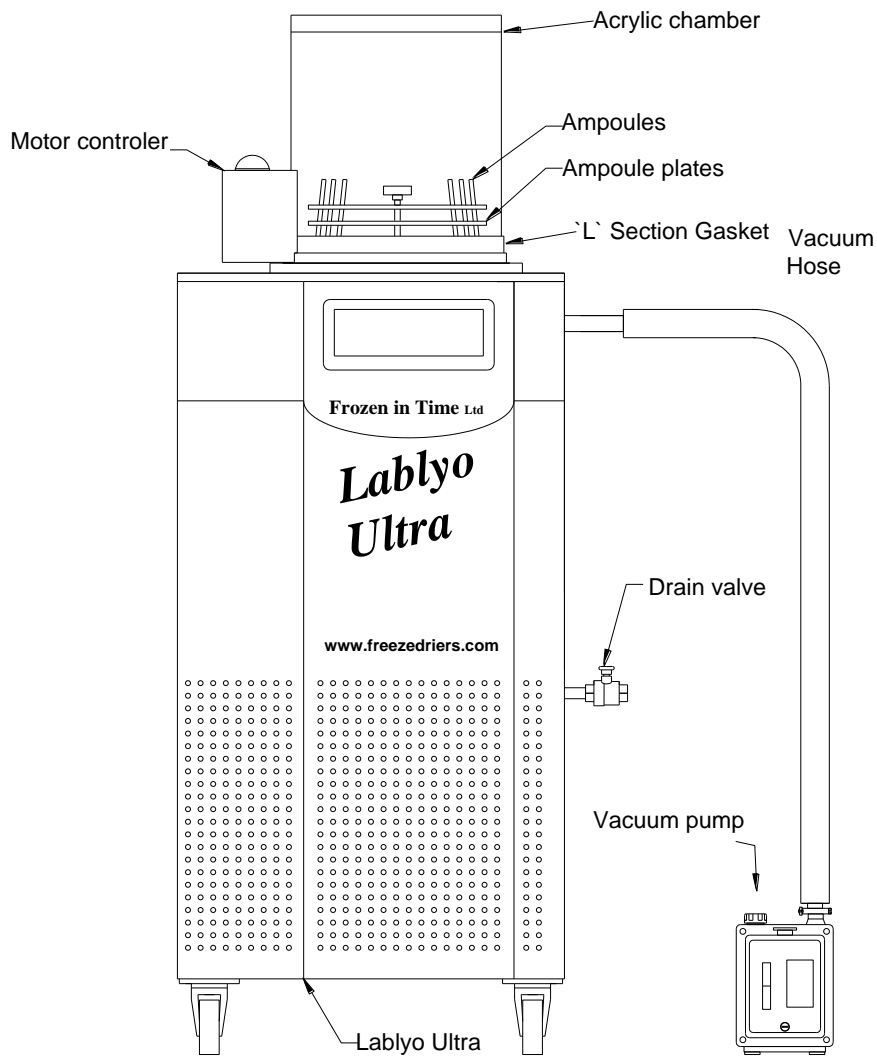
FIT/LYO/25/0 Lid with ampoule manifold 48-port

Additionally requires:

1 x 'L' section gasket

FIT/LYO/40/0

4.7.3. Spin freezer



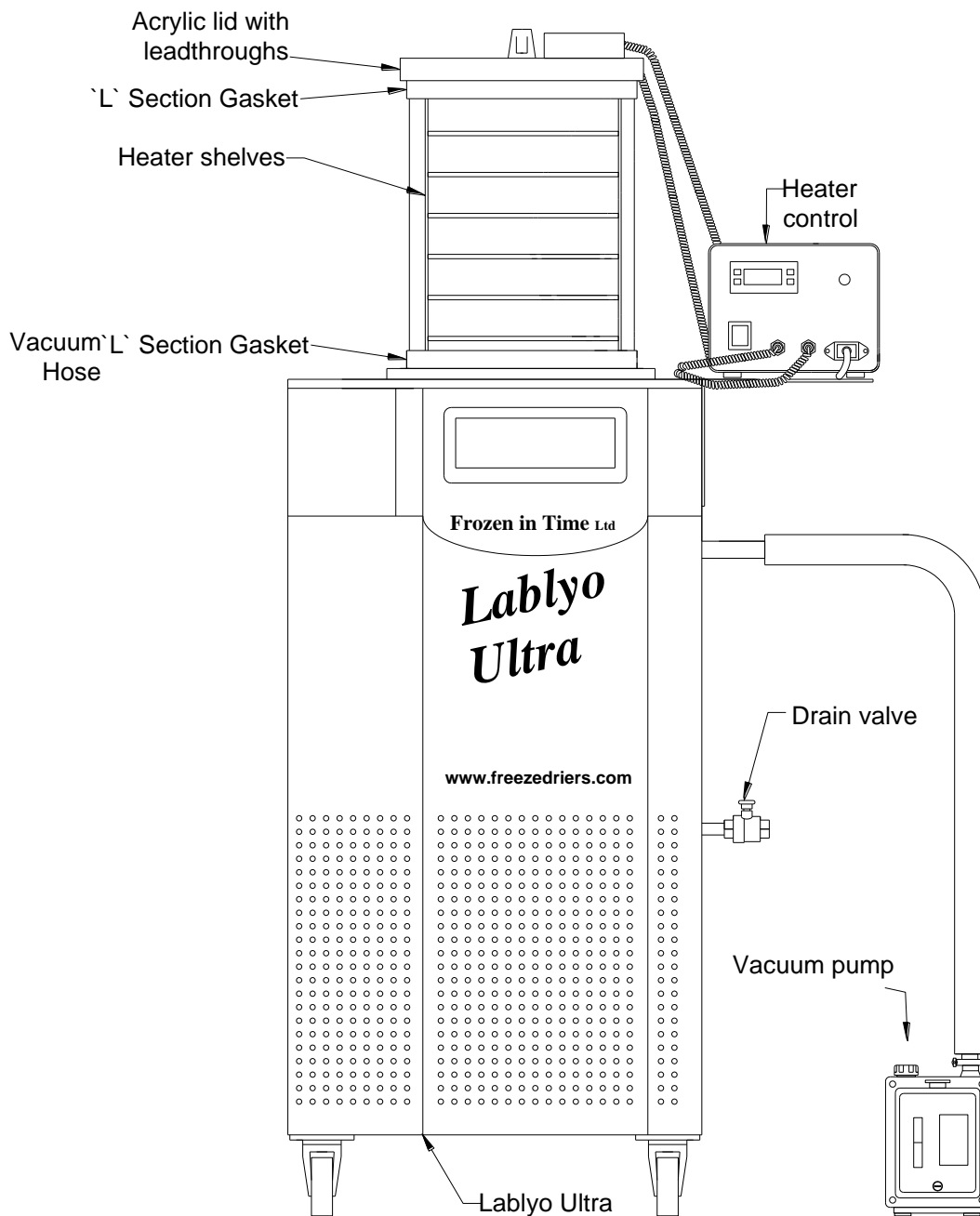
FIT/LYO/28/0 Spin Freeze with 1 x set carrier plates

Includes `L section gasket

Additionally options:

Extra Spin Freeze plates FIT/LYO/31/0

4.7.4. Heated shelves

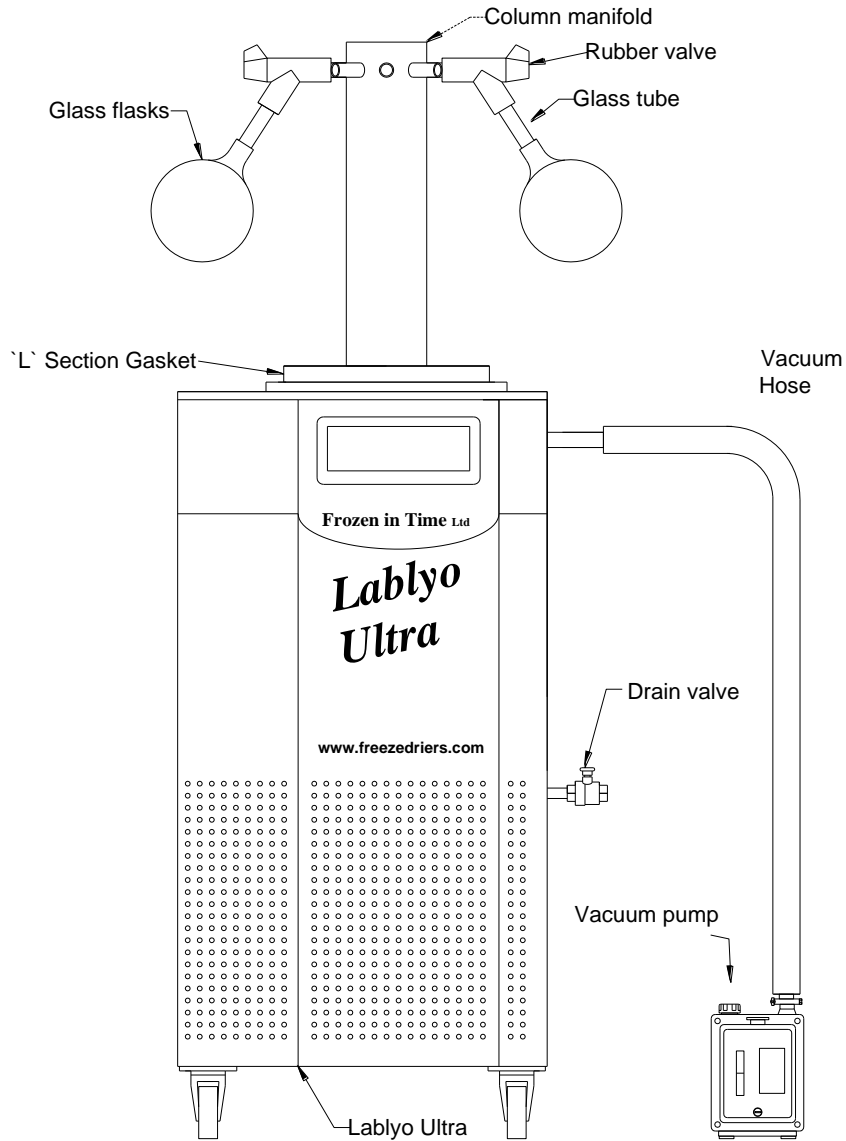


FIT/LYO/19/0 Heated Shelves, 6 shelves, temperature controlled

Additionally requires:

- | | |
|-------------------------|--------------|
| Acrylic drying chamber | FIT/LYO/01/0 |
| 2 x `L` section gaskets | FIT/LYO/40/0 |
| 6 x Trays 230mm | FIT/LYO/34/0 |

4.7.5. 8 port column manifold



FIT/LYO/13/0 **Column manifold, 8-port**

Additionally requires:

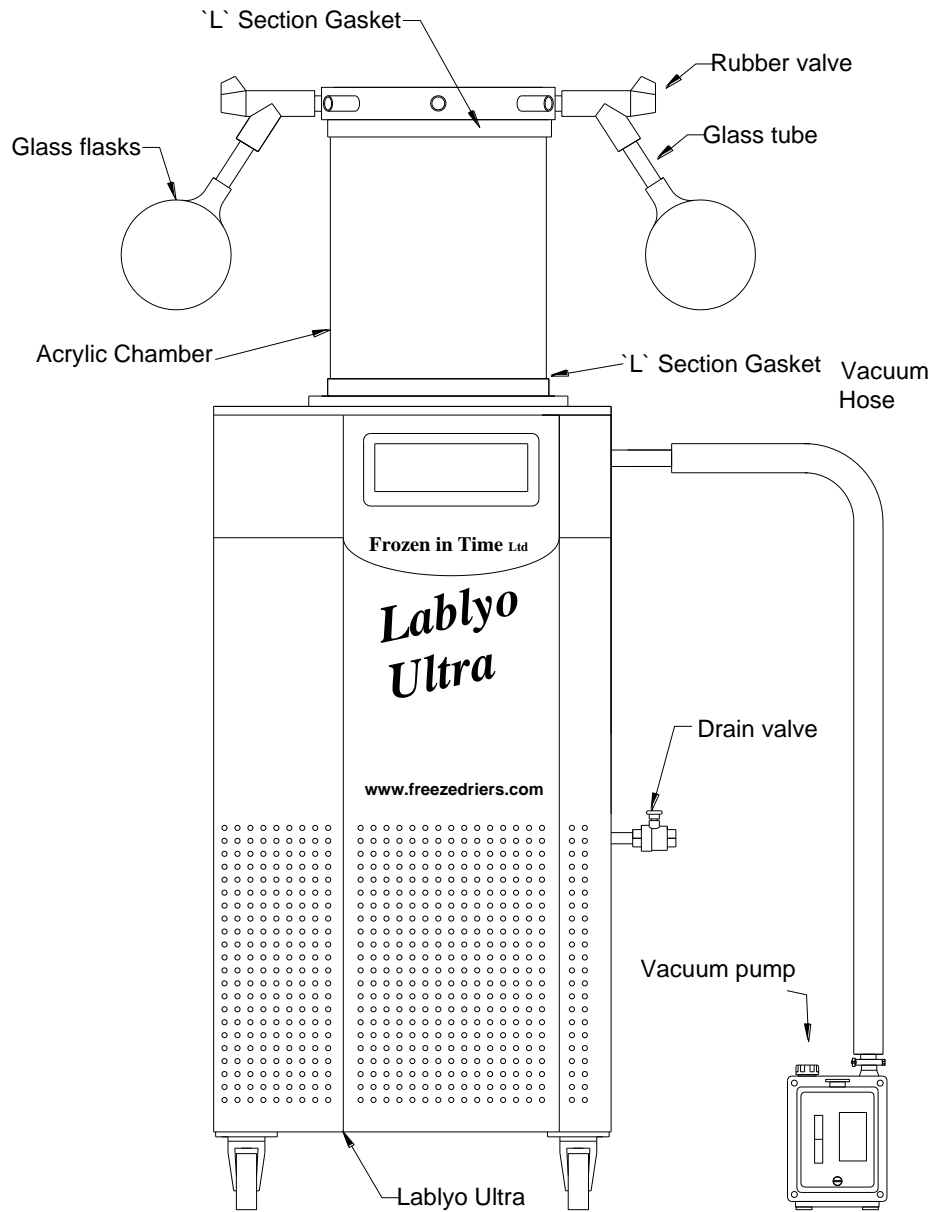
1 x `L` section gaskets

FIT/LYO/40/0

8 x Quick seal valves

FIT/LYO/43/0

4.7.6. 8 port lid manifold



FIT/LYO/6/0

Lid manifold 8-port

Additionally requires:

Acrylic drying chamber

FIT/LYO/01/0

2 x `L` section gaskets

FIT/LYO/40/0

8 x Quick seal valves

FIT/LYO/43/0

Additionally options:

6 shelf rack

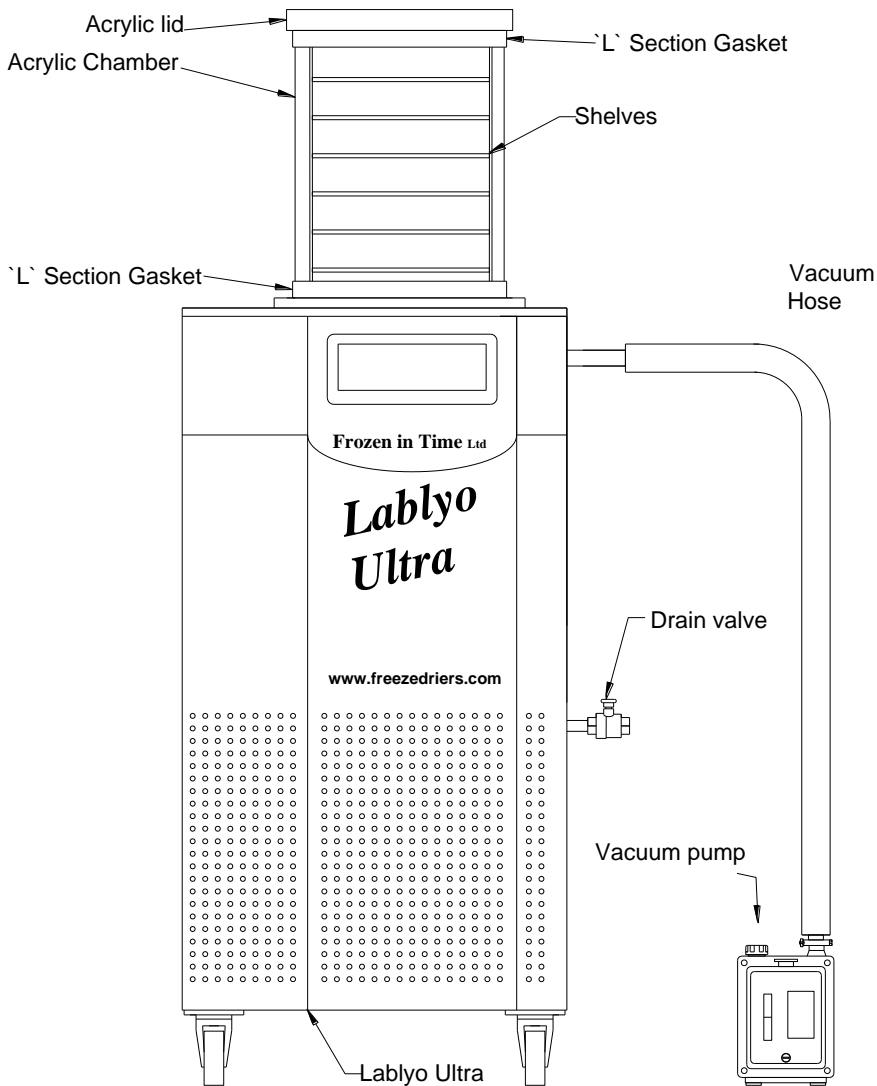
FIT/LYO/03/0

6 x Trays 230mm

FIT/LYO/34/0

To use the Lid manifold as a plain lid without valves it can be turned upside down.

4.7.7. 6 Shelf Rack



FIT/LYO/03/0 6 shelf rack for acrylic chamber (aluminum)

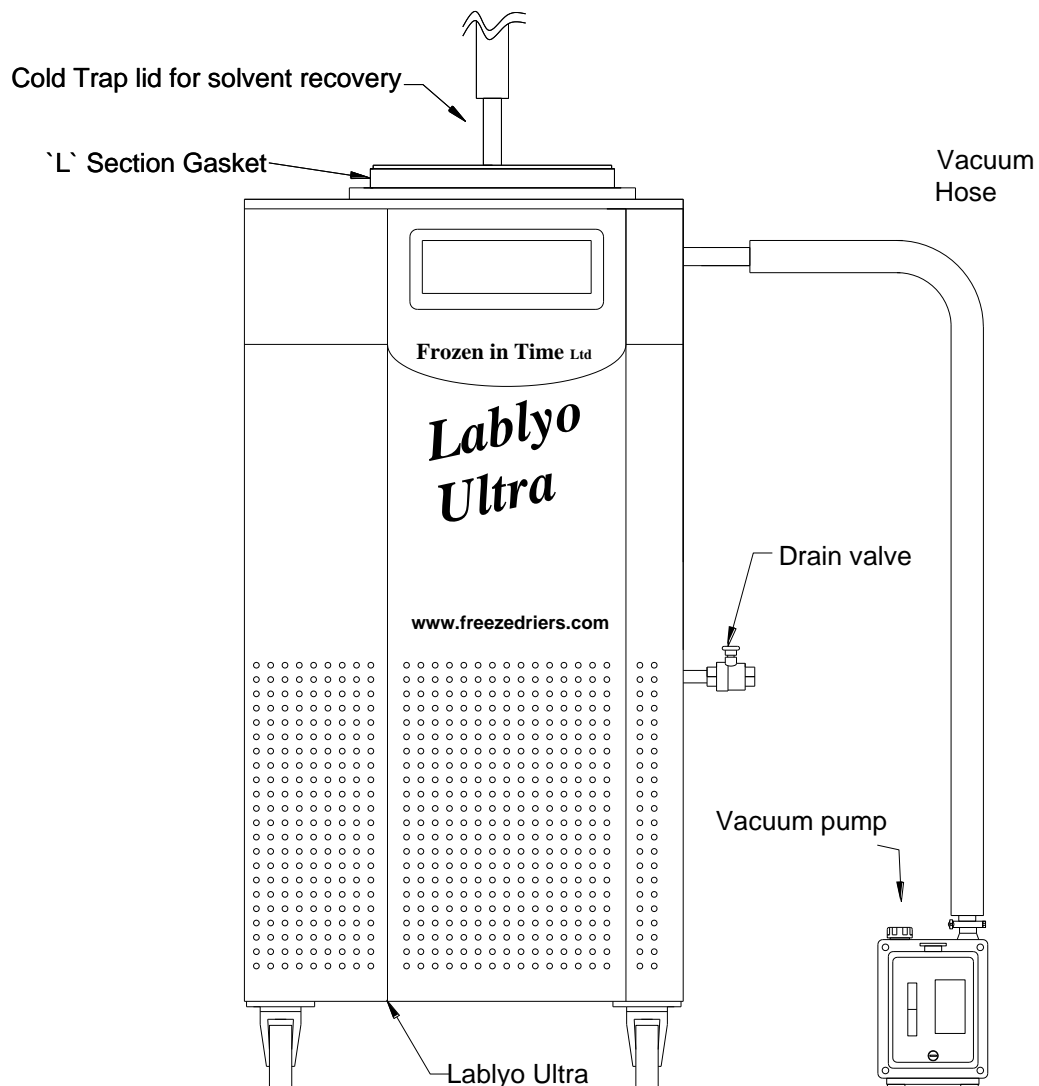
Additionally requires:

Acrylic drying chamber	FIT/LYO/01/0
2x `L` section gaskets	FIT/LYO/40/0
Lid for drying chamber	FIT/LYO/12/0

Additionally options:

Lid manifold 8-port	FIT/LYO/6/0
4 x Trays 230mm	FIT/LYO/34/0

4.7.8. The Lablyo Ultra as a standalone cold trap



FIT/LYO/9/0 Cold trap lid with hose for solvent recovery or vacuum pump protection

Additionally requires:

1 x `L` section gaskets

FIT/LYO/40/0

Lid can be made to order with any fittings to the customer's requirements

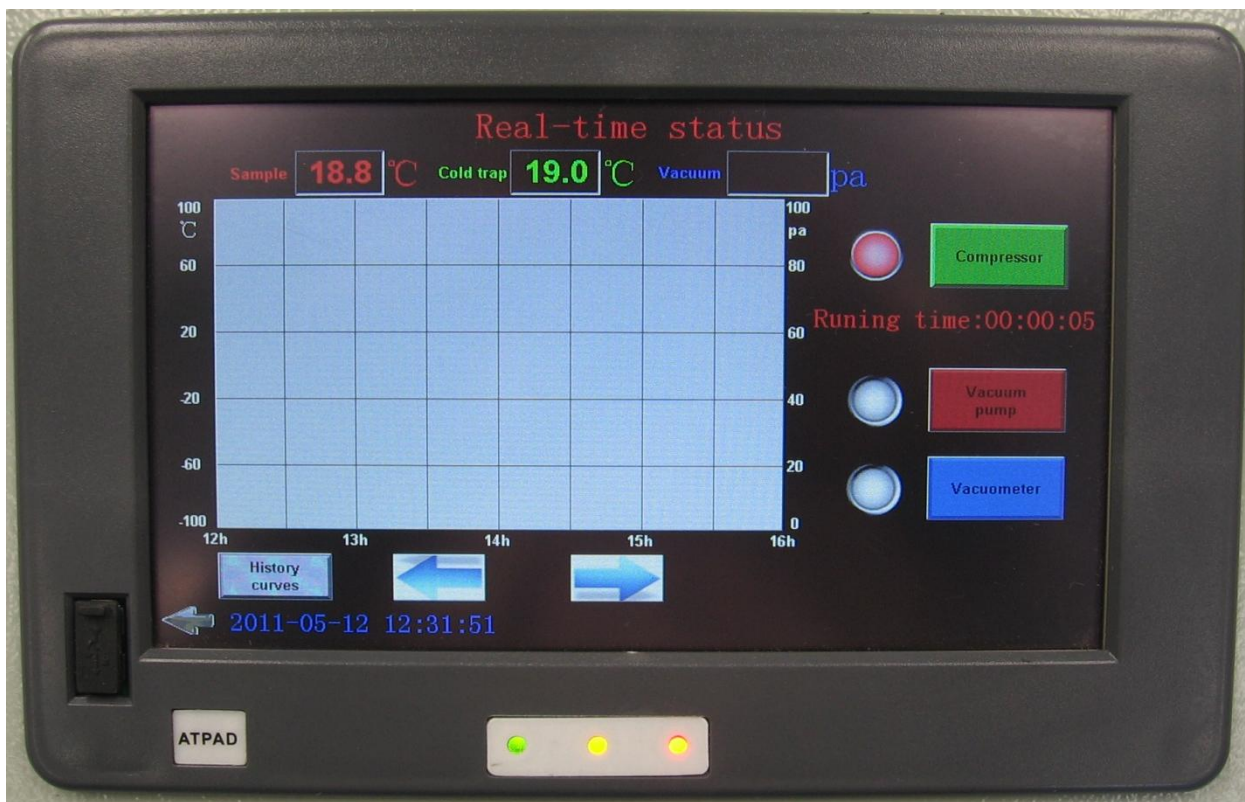
5. Using the Lablyo Ultra

5.1. Switching on the unit

Switch the Lablyo Ultra on at the power switch on the rear of the unit. The indicator light should now be on. The control panel should also now be active.

The screen shows a graph in the centre showing temperature on the right hand side in the Y axis.

- Then time along the X axis and vacuum level up the right hand side of the Y axis.
- The mobile sample temperature shows in the top left in red.
- The cold trap wall temperature is shown in the top middle in green.
- Vacuum level when switched on shows in the top right in blue. (switch is bottom right).



The main screens buttons are on the right, Compressor (green button), vacuum pump (red button) and Vacuum sensor (blue button)

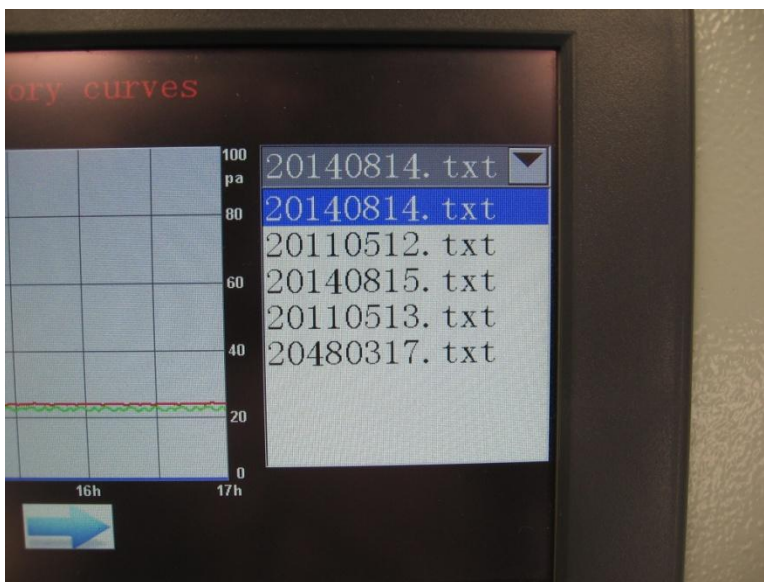
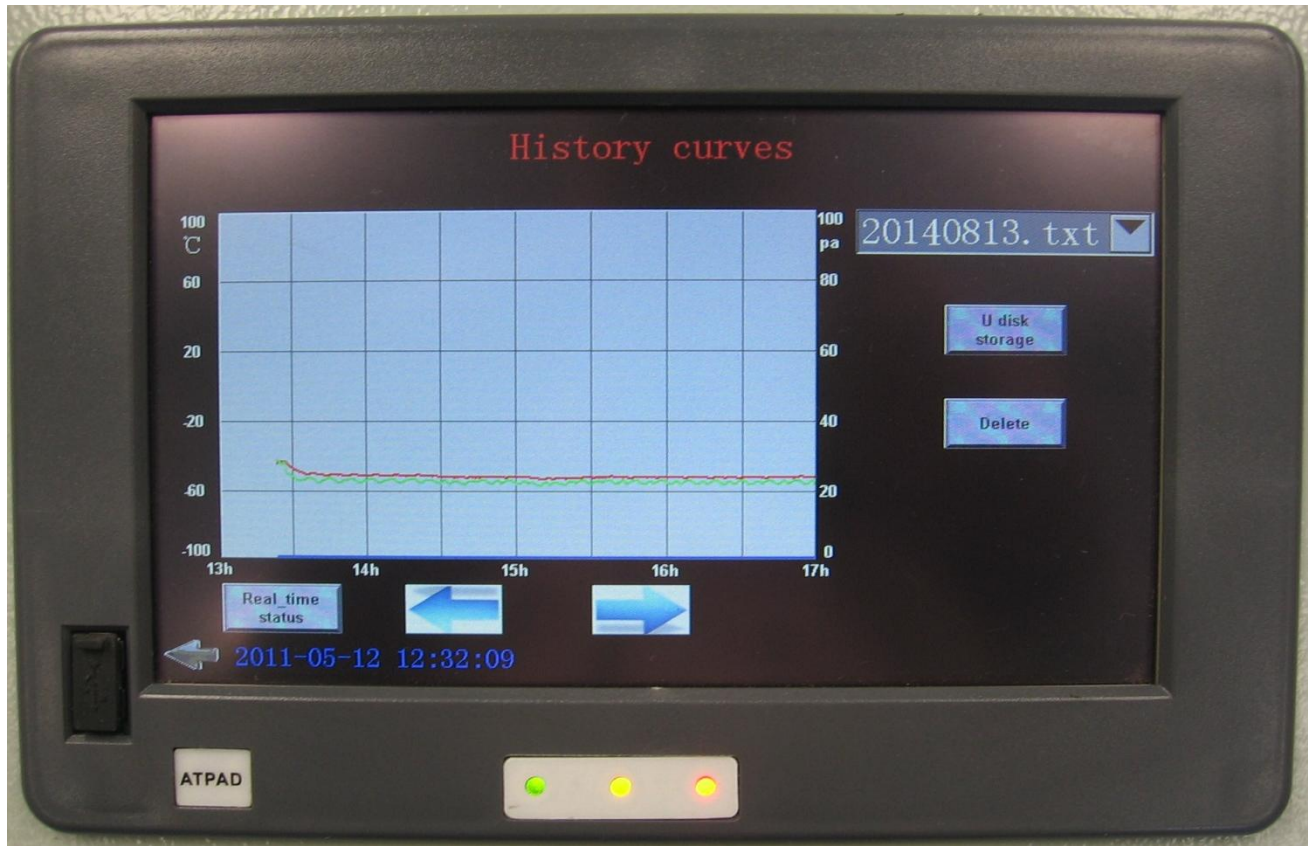
The graph traces will start on the current hour, not at 0h unless it is midnight. The traces will show the history of the Sample in red, the Cold trap in green and the Vacuum level in blue.

Scrolling back and forth is done by pressing the blue arrows.

The graph will just show up to the end of the day saving the data in a file in the History curves page.

A new graph will start for each day

In the history curves page you can select a file to look up They are saved as a file number that represents the year, month and day. Selected files can be opened on the screen as graphs or saved on a memory stick and opened on a computer as text files. Transfer to a computer is done via memory stick. To do this insert the memory stick and press U disk storage.



5.2. Starting the condenser chamber refrigeration

To start the condenser chamber press the green button labeled **Refrigeration** the red indicator light will illuminate and the refrigeration will start.

5.3. Defrost

Defrost is not part of the control panel. The red illuminated switch on the side activates the defrost. Defrost will always be overridden by the refrigeration. Defrost should only be switched on to defrost ice and it must then be switched off. However the defrost heater is self regulating and will not over heat.

5.4. Starting the vacuum Pump

To start the vacuum pump the condenser chamber must already be running and the temperature must be below -30°C. Press the **VACUUM PUMP** button the red indicator light will illuminate and the vacuum pump will start.

5.5. Vacuum display

The vacuum display is in pascals.
1 bar = 100,000 pascals
1mbar = 100 pascals

5.6. Temperature display

The temperature displays in °C this measures the wall temperature of the condenser chamber.

5.7. Drain valve

The drain valve should be closed whenever the vacuum pump is activated. It should not be opened while the vacuum pump is still switch on.
The drain valve is used to drain defrosted condensate from the condenser chamber. It is also used to admit air or inert gases into the condenser chamber to break the vacuum.

5.8. Vacuum connection

The IEC socket connection at the back of the unit is used to power a vacuum pump through the Lablyo Ultra making use of the control panel functions.

6. Accessories and part numbers

FIT/LYO/01/0 Acrylic drying chamber 300mm OD.

Additionally requires:

2x `L` section gaskets FIT/LYO/40/0

Required if using (with the Lablyo Ultra) :

6 shelf rack FIT/LYO/03/0
Heated shelves FIT/LYO/19/0
Lid manifold FIT/LYO/6/0
Closing device FIT/LYO/22/0

FIT/LYO/03/0 6 shelf rack for acrylic chamber (aluminum)

Additionally requires:

Acrylic drying chamber FIT/LYO/01/0
2x `L` section gaskets FIT/LYO/40/0

FIT/LYO/6/0 Lid manifold 8-port

Additionally requires:

Acrylic drying chamber FIT/LYO/01/0
2 x `L` section gaskets FIT/LYO/40/0
8 x Quick seal valves FIT/LYO/43/0

FIT/LYO/9/0 Cold trap lid with hose for solvent recovery or vacuum pump protection

Additionally requires:

1 x `L` section gaskets FIT/LYO/40/0

FIT/LYO/12/0 Lid for drying chamber plain 320mm diameter x 20mm thick

Additionally requires:

Acrylic drying chamber FIT/LYO/01/0
2 x `L` section gaskets FIT/LYO/40/0

FIT/LYO/13/0 Column manifold, 8-port

Additionally requires:

1 x `L` section gaskets FIT/LYO/40/0
8 x Quick seal valves FIT/LYO/43/0

FIT/LYO/16/0 Column manifold, 16-port

Additionally requires:

1 x `L` section gaskets FIT/LYO/40/0
16 x Quick seal valves FIT/LYO/43/0

FIT/LYO/19/0 **Heated Shelves, 6 shelves, temperature controlled**

Additionally requires:

Acrylic drying chamber FIT/LYO/01/0
2 x `L` section gaskets FIT/LYO/40/0

FIT/LYO/22/0 **Vial Closing Device**

Additionally requires:

Acrylic drying chamber FIT/LYO/01/0
2 x `L` section gaskets FIT/LYO/40/0

FIT/LYO/25/0 **Lid with ampoule manifold 48-port**

Additionally requires:

1 x `L` section gasket FIT/LYO/40/0

FIT/LYO/28/0 **Spin Freeze with 1 x set carrier plates**

Additionally options:

1 x Extra Spin Freeze plates FIT/LYO/31/0

FIT/LYO/34/0 **Trays 230mm for vials or tray drying x1**

Additional option for the:

6 shelf rack FIT/LYO/03/0
Heated shelves FIT/LYO/19/0
Closing device FIT/LYO/22/0

FIT/LYO/37/0 **Vacuum hose 1m for vacuum pump**

Additional option for the:

Vacuum Pump FIT/LYO/59/0

FIT/LYO/40/0 **`L` section gaskets for accessories**

Additional option for the:

Column manifold, 8-port FIT/LYO/13/0
Column manifold, 16-port FIT/LYO/16/0
Lid manifold 8-port FIT/LYO/25/0
Acrylic drying chamber FIT/LYO/01/0
Lid for drying chamber FIT/LYO/12/0

FIT/LYO/43/0 **Quick seal valves**

Additional option for the:

Column manifold, 8-port FIT/LYO/13/0
Column manifold, 16-port FIT/LYO/16/0
Lid manifold 8-port FIT/LYO/16/0

7. Troubleshooting

7.1. Power failure

The refrigeration and vacuum pump will restart after a power failure. In the event of a power failure in the drying phase, the batch may become unusable. Whether the batch can be saved or not depends on the drying phase the product was in when the power failure occurred. If power returns within a few minutes then it is unlikely that any damage will have occurred. It is important to distinguish between the primary drying phase and the secondary drying phase. The product is in the secondary drying phase if the residual moisture has reached approximately 5 %. Below this value, the product is generally not damaged by a power failure.

If the product is in the primary drying phase, we recommend removing it for refreezing.

It is advisable to defrost the condenser chamber at this point.

If the product is considered to be still usable then it can be placed back in the freeze drier and the process can be started again.

7.2. Insufficient vacuum

7.1.1. Easy initial checks

Check that the drain valve is closed. Remove the accessory and seal the top of the condenser chamber with a blank lid and a lightly greased `L` section gasket.

If the lid now seals it indicates that there is a leak in the accessory.

If the lid does not seal then the drain valve, vacuum hose or vacuum pump is at fault.

Check that the vacuum pump is running and that there is suction. If there is, check that the hose push on connections between Lablyo Ultra and vacuum pump are tight. Make sure that the drain valve is not leaking by checking for suction when it is closed.

If the accessory is leaking examine every port and push on fitting. Lightly grease any sealing parts and press down on chamber or manifold in order to assist the sealing gaskets.

7.1.2. Checks to make using the vacuum readout

The following steps require that there is no ice or water inside the condenser chamber. Dry the chamber with a cloth. To test the Lablyo Ultra, connected to the vacuum pump and put a blank lid and seal on the mouth of the chamber. It will not achieve the same level of vacuum as a direct reading off the pump but it should probably be lower than 0.1 mbar. The next thing to do would be to fit the accessory if it is a manifold with rubber valves then these should all be closed.

If it is a 48 port manifold then all 48 ports need to be blanked off or fit with ampoules.

All accessories with acrylic chambers need to be assembled complete.

To check the vacuum pump directly it is necessary to use a separate vacuum sensor. Make sure that the vacuum pump is warm and there should be a vacuum reading of 0.04 mbar or less.

7.3. Unit does not work

If the indicator light does not work and the refrigeration system is not running after the mains switch has been activated, the following tests must be performed:

Check that the Lablyo Ultra is plugged in.

Check the breakers or fuses for the circuit.

Check the plug fuse.

7.4. Insufficient ice condenser temperature

The refrigeration will stop if the ambient temperature is too high or the airflow is restricted to the heat exchanger. Once the permissible operating conditions are reached again the refrigeration unit is switched on automatically via the motor protection switch or via the pressure switch.

The minimum ice condenser temperature of approximately -55°C or -85°C is reached when the ice condenser is not under load. Sufficient air circulation is very important. Do not place any objects behind the unit!

8. Maintenance

8.1. Ice condenser chamber

Before each start-up, ensure that all water residues have been removed from the ice condenser chamber. If necessary, wipe the ice condenser chamber dry. Before every drying process it is recommended to open and close the drain valve.

8.2. Heat exchanger

The refrigeration heat exchanger is used to cool the refrigerant compressed by the refrigeration unit. The heat exchanger is located at the back of the unit and should be checked for dust or dirt residues every few months. It must be cleaned whenever necessary. The heat exchanger can be cleaned best by brushing, by using a vacuum cleaner from the outside or by using compressed air from inside of the unit. Excessive build up on the heat exchanger leads to a decrease in performance and may cause a failure of the unit!

8.3. Rubber valves and seals

Special attention must be paid to the rubber valves. If the valves are stiff, they must be dismantled, cleaned, slightly greased with vacuum grease and reassembled.

8.4. Vacuum pump

For the maintenance of the vacuum pump, please refer to the separate operating manual. Additionally, we would like to emphasize the following points: The oil level of the vacuum pump must be regularly checked at the sight glass (in case of continuous operation at least once a day). Top up oil to the required level via the oil inlet. Due to possible operation with gas ballast, oil consumption cannot be avoided. For topping-up see the operating manual of the pump. The oil change should always be carried out with warm pump.

8.5. Exhaust filter

If the unit is equipped with an exhaust filter (necessary if the exhaust gases cannot be extracted into the open air or into a vent), take care that the condensate in the filter does not rise too high.

8.6. Cleaning

8.6.1. Cleaning the freeze dryer

Use soapy water or other water-soluble, mild cleaning agents to clean the freeze dryer. Avoid corrosive and aggressive substances. Do not use alkaline solutions or solvents or agents with abrasive particles. Remove product residues from the ice condenser chamber using a cloth. It is recommended to open the lid of the condenser chamber when the freeze dryer is not in use so that moisture can evaporate.

If there is the risk of toxic, radioactive or pathogenic contamination, special safety measures must be considered and adhered to.

8.6.2. Cleaning the accessories

When cleaning accessories, special safety measures must be taken as these are measures to ensure operational safety and reliability. Cleaning of accessories should be carried away from the freeze dryer once a week or preferably after every use. Aluminium accessories are particularly prone to corrosion.

If there is the risk of toxic, radioactive or pathogenic contamination, special safety measures must be considered and adhered to.

8.6.3. Maintenance of the air admittance/drain valve

Special attention must be paid to the air admittance/drain valve. If residues from previous drying processes deposit on it, the freeze dryer may not achieve correct vacuum levels. Therefore, take care that no product or other residues will get into the valve.

8.6.4. Disinfecting the drying chamber, the lid and the accessories

Check compatibility with the lid and the drying chamber. The freeze dryers and the accessories are made of different materials. A possible incompatibility must be considered. For autoclaving, the temperature stability of the individual materials must be taken into consideration. Please consult us if in doubt.

If dangerous materials are used, the freeze dryer and the accessories must be disinfected.

8.7. Checks by the operator

The operator has to ensure that the important parts of the freeze dryer that are necessary for safety are not damaged. This especially refers to:

- Lid or drying chamber
- Seals
- Oil level of vacuum pump
- Accessories, especially changes like corrosion, wear and abrasion of material etc.
- PAT test must be carried out annually.

9. IQ/OQ Qualification

This Qualification Protocol is solely intended to be used with Lablyo Ultra freeze driers which are new or relocated.

It is written to assist the end-user in validation of predetermined specifications. The protocol begins with planning the site for the piece of equipment and therefore is of value prior to receipt of delivery.

The use of this document does not replace the need for the Lablyo Ultra User's Manual and is in this case attached. Information within the User's Manual is required to complete this IQ/OQ

Protocol. If the manual has been misplaced, copies can be obtained from the manufacturer or down-loaded from their website, www.freezedriers.com

9.1. Installation Qualification

Step	Description	Specification or Acceptance Criteria	Result	
1	Site Planning			
1a	Space Requirements	Refer to 1.3 Technical data of freeze dryer in User's Manual for dimensions of the model you have chosen. Has adequate floor or counter space been provided for placement of the equipment? (A minimum of 100mm is required between the back of a freeze dryer and the wall and between the sides and the walls for proper airflow through the refrigeration system.)	Y	N
1b	Electrical Service	Refer to the User's Manual for electrical requirements. Are services available for the equipment to be connected to an electrical circuit of adequate size and the proper voltage?	Y	N
1c	Vacuum Pump	Refer to the User's Manual. Have accommodations been made to provide a suitable vacuum pump? It must be capable of: at least 100 Liters/min With an inlet fitting suitable for a ¾-inch ID vacuum hose on one end and a KF25 on the other. It must have the same voltage rating as the freeze dryer? And have the means to be powered by a IEC connection. (An oil mist eliminator exhaust filter is recommended.)	Y	N
1d	Accessories	Freeze drying chambers or manifolds are not included with the freeze dryer. Has an appropriate accessory been purchased to complete the required freeze drying process.	Y N/A	N

2		Prior to operation		
2a	Damage Claims	Have the delivered products been inspected for any signs of damage that may have occurred while in transit? Keep packaging materials until inspection is complete. WARNING: Do not attempt to pull a vacuum on a freeze dryer with any damage to any of the accessories manifolds/chambers etc. Implosion and potential for injury can occur. If damaged contact Frozen in Time 01347 878158 or the distributor from whom it was purchased	Y	N
2b	Handling Solvents	Has the Safety Officer or equivalent reviewed the safe handling and disposal of solvents trapped as well as used vacuum pump oil?	Y N/A	N
2c	Manifold Installation	Place the desired drying chamber or manifold on to the top of the Lablyo Ultra condenser chamber flange. Do you have the correct number and size of `L` section gaskets to make the vacuum seal?	Y N/A	N
2d	Vacuum Pump Installation	Install the vacuum pump per the User's Manual. Is the pump attached to the vacuum port at the top of the chamber with the correct rubber hose and clamps provided?	Y	N
2e	Vacuum Pump Electrical	Is the vacuum pump plugged into the rear of the freeze dryer and the pump's power switch turned to the ON position?	Y	N
	Vacuum Ballast	The vacuum pump's ballast should remain closed. Is the pump's ballast closed?	Y	N
	Electrical Connection	Plug the freeze dryer into a dedicated electrical outlet. Has the electrical service been verified to be adequate in size and voltage? (The ID plate on the rear of the freeze dryer has the electrical requirements.)	Y	N
2e	Electrical Grounding	Has the ground on the electrical service been verified?	Y	N

9.2. Operational Qualification

Step	Description	Specification or Acceptance Criteria	Result	
1	Freeze Drier			
1a	Refrigeration	With the freeze dryer system at ambient temperature, turn the Main Power Switch ON. Press the button labeled “Refrigeration.” Does the refrigeration system start? Record the time it started. _____	Y	N
1b	Power to Vacuum Pump	Press the button labeled “Vacuum Pump.” Does the vacuum pump start? Record the time it started. _____	Y	N
1c	Refrigeration Effectiveness	Lablyo Ultra freeze driers does the condenser chamber temperature reach -47°C in less than 40 minutes? (With the system under vacuum and 21°C ambient temperature.) NOTE: Freeze Dryers are tested to -50°C or in the factory. Conditions may vary in the field resulting in warmer acceptance temperatures.	Y	N
1d	Verify Displayed Temperature	The temperature indicated on the LCD display is measured on the collector coil. There is a mobile probe that hangs free inside as well. The values were calibrated at the factory by correlating its reading with that of a reference gauge attached to a sensors. Does the display correlate to the reference Gauge $\pm 2^{\circ}\text{C}$? Ref. Instrument? _____ If the temperature does not correlate, contact Frozen in Time 01347 878158	Y N/A	N
1e	Vacuum Leaks	Verify that the system is leak-free by continuously running the vacuum pump with the refrigeration system ON. The rate the freeze dryer without samples achieves a low level of vacuum, (less than 13 Pa), it is dependent upon many factors: Inside volume & surface area of the system. Cleanliness or cleaners used on interior. Condition & size of the vacuum pump. Period of time the parts have been exposed to environmental conditions. Based on the freeze dryer’s displayed vacuum level, the freeze dryer should reach its lowest level in less than 18 hours. If not, refer to Vacuum Troubleshooting Guide in the User’s Manual. Does the system reach a displayed vacuum level of less than 6Pa in 18 hours?	Y	N
1f	Temperature Graph Display	The temperature graph traces on the control panel is a quick reference of the temperatures. Verify these graphs are operating properly. Compared to the readouts.	Y	N

1g	Vacuum Wave Display	The control panel's vacuum trace is a quick reference of vacuum level. Verify this is operating properly. Compared to the readouts.	Y N/A	N
1h	Verify Displayed Vacuum	<p>The vacuum level indicated on the LCD display is measured between on the condenser chamber. The value was calibrated at the factory by correlating its reading with that of a reference gauge. The calibration was performed at a very low level, approximately 10Pa.</p> <p>NOTICE: Factory calibration was performed using a precision Active Piranni Gauge calibrated to a Capacitance Manometer standard. Despite the system's calibration and repeatability, the readings taken at such a low level of vacuum should only be considered as a verification of a leak-free system. Vacuum swings can be attributed to contamination of surfaces, which could take days to outgas. Adjustments are discouraged. Before any adjustments are made to the factory calibration of the vacuum measurement, answer positively to these questions:</p> <p>1) Is the vacuum standard being used to verify the freeze dryer accepted by the organization to be precise and has it organization to be precise and has it been calibrated recently?</p> <p>2) Is the level of accuracy we are attempting to reach pertinent to the freeze drying applications? Does the vacuum display correlate to the reference gauge?</p> <p>Ref. Instrument? _____</p> <p>If vacuum is to be calibrated, contact Labconco Product Service for calibration procedure.</p>	Y N/A	N
1i	Defrost	<p>With the refrigeration switched OFF, press the "defrost" button ON.</p> <p>Does the condenser chamber become warm to the touch?</p>	Y N/A	N

2	Routine Maintenance	Below are helpful hints to be included in the organization's preventive maintenance plan.		
2a	Vacuum Grease	Vacuum grease should be applied to rubber components as required. In general, vacuum grease should be the first step in trouble shooting vacuum leaks. Thin layers of grease are adequate for all seals. Only use grease specially formulated for low vacuum service. Is vacuum grease readily available and documented? Type of grease used? _____	Y	N
2b	Vacuum Pump Oil	The vacuum pump oil should be changed as needed. Change oil that appears cloudy or discolored. At a minimum, oil should be changed every 1000 hours of service. Has there been a preventive maintenance plan established for the vacuum pump? Type of oil to be used? _____	Y	N

3	Personnel Training			
3a	User Training Related to Equipment	Have personnel that will use the Lablyo Ultra freeze drier been adequately trained? Are personnel familiar with: All the buttons and displays on the front; Collector capacity limits before defrosting; Defrosting and draining methods; Safe handling of solvents drained; The use of vacuum grease; Opening, closing and venting sample valves; Cleaning of the freeze dryer and neutralization of acids?	Y	N
3b	User's Manual	Are the personnel who are to use or maintain the Lablyo Ultra able to locate the User's Manual for the machine?	Y	N

9.3. Summary

Lablyo Ultra freeze-drier IQ/OQ Document

Equipment Location _____

FreeZone Ser. No. _____ Model No. _____

User Protocol _____ Revision (or Date published) _____

Contact (print name): _____

Title: _____

Review the “Response” columns for answers of “NO.” Use the area below to describe the deficiency or unacceptable results. Those deficiencies are to be followed with an instruction for “Corrective Actions.” Once acceptable results are obtained, the deficiency is “accepted” by signing the Corrective Action.

Step	Deficiency followed by Corrective Action	Signature