

Frozen in Time Ltd

Manufacturers of Freeze Drying Machines and Vacuum Cold traps

Operating Manual

HS-2 & HSL-2



Operating Manual

HS-2 & HSL-2

Order Number:

Serial Number:

In case of enquiries please state the above

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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 to dry some inorganic products.

1.2. Technical data of freeze dryer

HS-2 & HSL-2

	HS-2	HSL-2
Overall Dimensions W x D x H cm	95 x 100 x 189	95 x 100 x 189
Specimen Chamber Diameter x Length cm	65 x 65	65 x 65
Specimen and Condenser Chamber Construction	Polished 316L Stainless Steel	Polished 316L Stainless Steel
Tray Area m²	0.9	0.9
Number of shelves	5+1	5+1
Shelf Size W x D in cm	40 x 50	40 x 50
Condenser Chamber Temperature	-55 °C	-85 °C
Shelf Temperature Range °C	-40 to +60 °C	-65 to +60 °C
Condenser chamber Defrost	Hot Gas	Hot Gas & Electric
Chamber Doors	Clear Acrylic	Clear Acrylic
Condenser Ice Capacity	25kg (15kg/24h)	25kg (15kg/24h)
Refrigerant	R-507	R-507 & R-23
Vacuum Pump	Woosung W2V30	Woosung W2V20
Refrigeration HP	2 x 2.0hp	2 x 2.0hp
Isolation Valve Ø cm	Butterfly 15	Butterfly 15
Power Requirements	3ph 415v	3ph 415v

1.3. Safety instructions

1.3.1. Disconnect the electricity supply before removing panels

The freeze drier must be securely isolated or unplugged from the mains supply before the panels are removed or any maintenance work is undertaken.

1.3.2. Solvents

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

1.3.3. Cleaning and Maintenance of the Unit

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

1.3.4. 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.

1.3.5. Transporting

The **HS-2** & **HSL-2** are on castor wheels and can be moved from one location to another if necessary

1.4. The HS-2 & HSL-2 should not be used when:

1. It is not properly installed.
2. Panels are missing.
3. The operator is not authorized or trained
4. Highly corrosive or solvent substances are present.
5. In hazardous or dangerous locations.
6. The products are explosive or highly flammable.
7. The products are Infectious, toxic, pathogenic or radioactive unless in suitable vessels and in accordance with the relevant safety data.

2. Description of the Freeze Drying Processes

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 vapour state. The frozen product is dried under vacuum without thawing. The condenser chamber works as a cryogenic pump as it takes large volumes of vapour and condenses a small amount of ice. The vacuum pump is only intended to remove the air from the drying chamber but not the vapour. In order to start the sublimation process, energy must be supplied to the product. This energy source comes from the heated shelves.

Primary drying removes the most of the water from the product.

Secondary drying removes the last traces of water by means of deep vacuum.

The main components of a freeze dryer are:

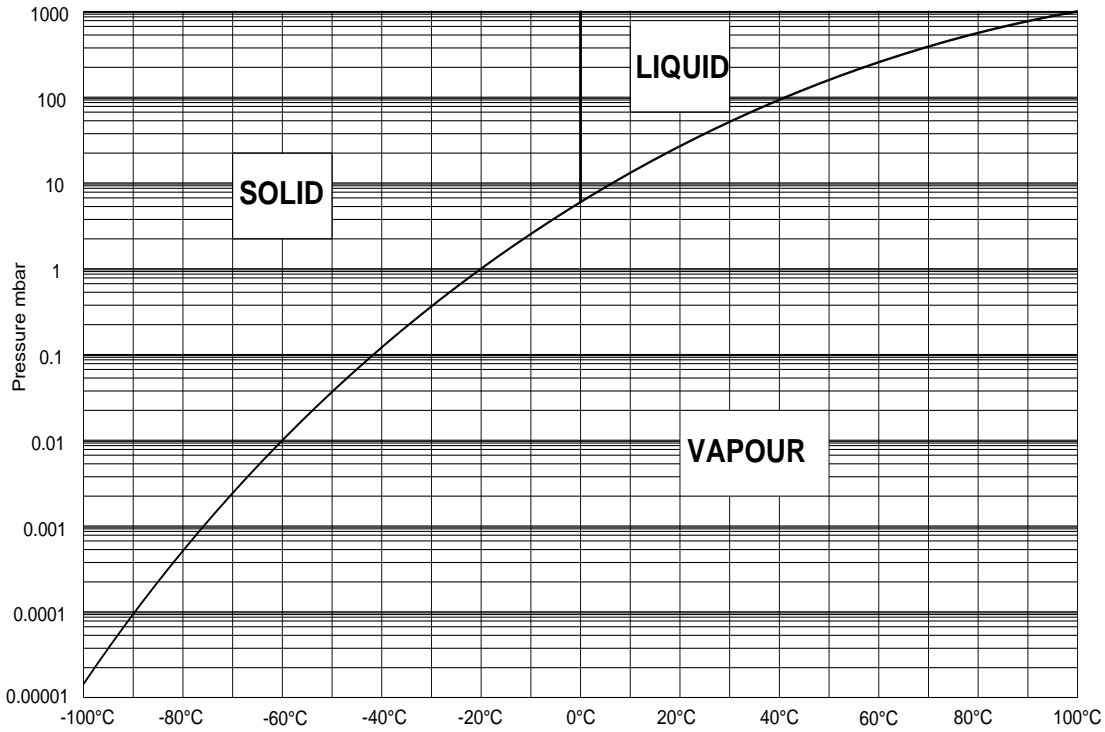
- Vacuum drying chamber, probably with heated shelves and a vial closing device
- Vacuum pump to evacuate air.
- Ice condenser with temperature of -55°C or -85°C to condense water vapour.

Sublimation

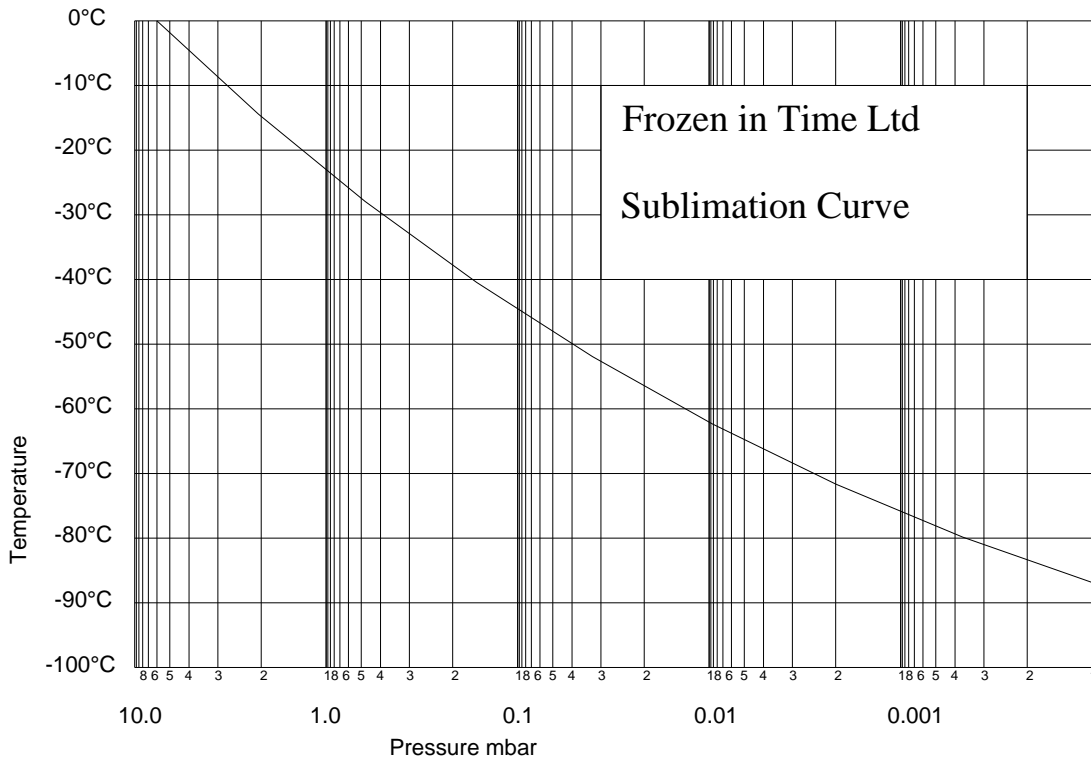
The principle of sublimation is briefly explained using the phase diagram of solid liquid and vapour. If the pressure is higher than 6.1 mbar, water can exist in all three phases (solid, liquid, vapour) when the temperature is lowered or raised. At 6.1 mbar and 0°C all three lines meet, this is called 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.

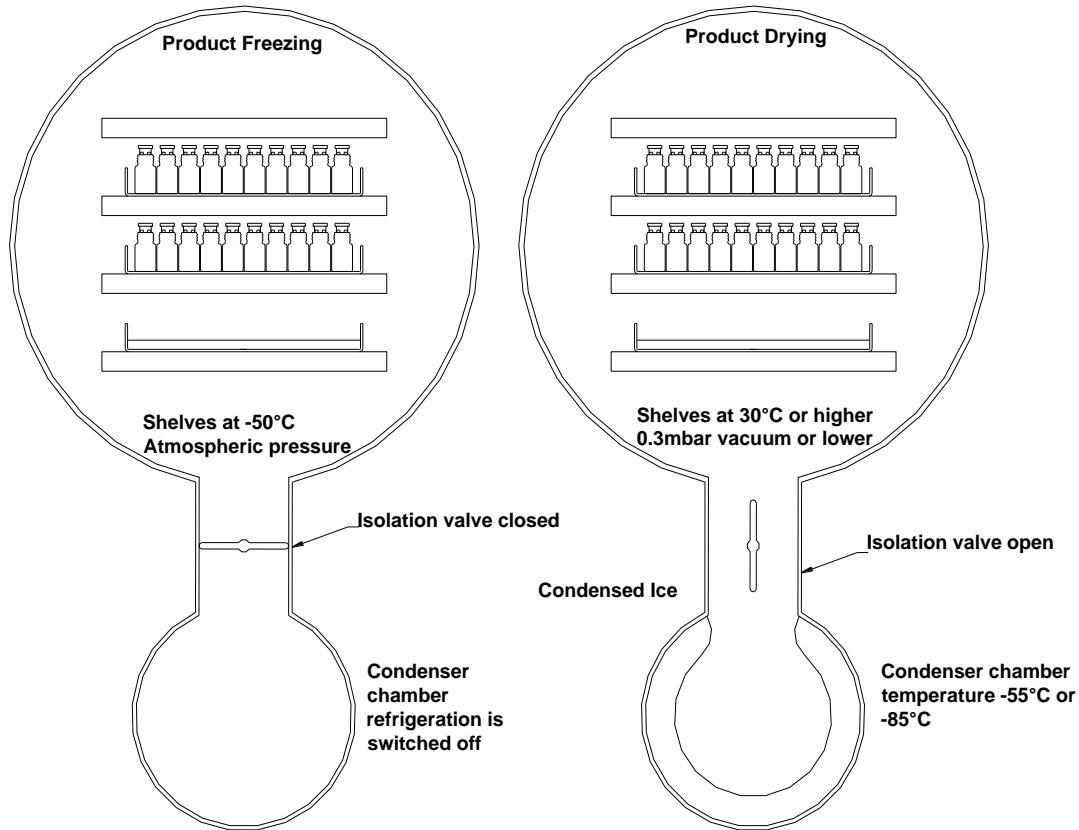
The sublimation curve shows the vapour pressure of water as affected by temperature and pressure.

Phase diagram for solid, liquid and vapour



Vapour pressure of ice





Cross sectional diagram of the freeze driers chambers during **Product freezing** and during **Product drying**.

Product Freezing

The Isolation valve is closed and the product is frozen by the low temperature of the shelves.

Product drying

The condenser is frozen to its minimum temperature. As the temperature goes below approximately -30°C the vacuum pump can be switched on. The isolation valve must be opened and the vacuum is created in both chambers. When the vacuum is sufficient the heating can be applied to the shelves. Sublimation will cause the products on the shelves to dry. Deposition will occur in the condenser chamber as the sublimated vapour re-freezes.

2.2. Freezing the product.

Product can be frozen directly on the shelves of the **HS-2 & HSL-2**. This is the usual option for product to be freeze dried in trays or vials. The product is placed on the shelves and the shelves are cooled down to the desired temperature. The freezing can be done in stages, at a controlled rate or just as quickly as possible.

2.3. Primary drying

The condenser drops to its operating temperature.

The isolation valve is open

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.

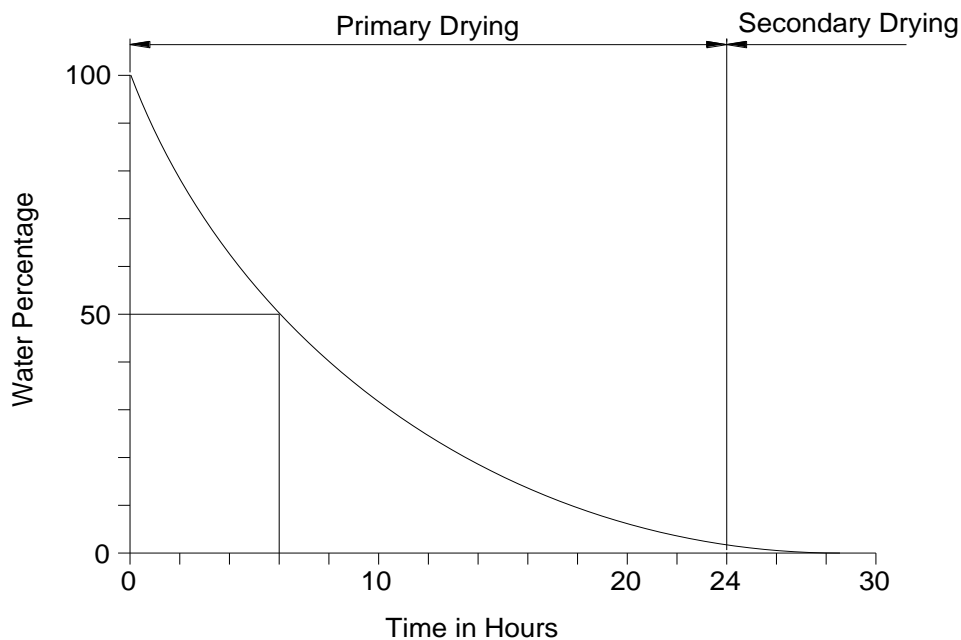
During primary drying the moisture is removed by sublimation and during secondary drying the bound moisture is removed by desorption. The recommended vacuum pump should reach with open gas ballast valve, a vacuum level lower than the relevant water vapour pressure.

Shelf temperatures can be raised in stages, at a controlled rate or set to maximum for fastest heat transfer. The heat input must of course not be enough to damage or melt the product. Too much heat will increase the amount of vapours to a point where the vacuum level diminishes to above the eutectic point of the product.

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 approximately 50% of the water content is removed. During the next quarter of the primary drying phase approximately 50% of the remaining water content is removed. 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 vapour pressure curve the shorter the drying time.

Facts regarding ice in a vacuum:

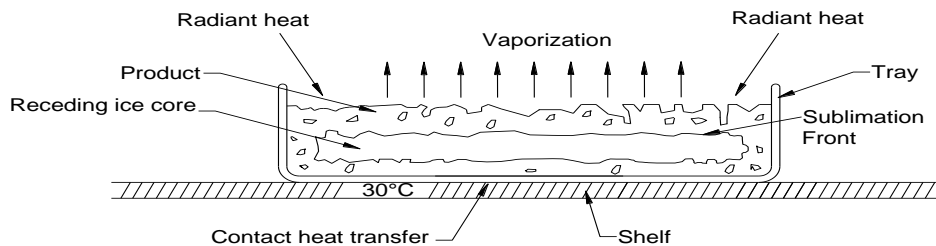
- 1.0 gram of ice at;
- 1.0 mbar assumes a volume of 1 m³ vapour
- 0.1 mbar assumes a volume of 10 m³ vapour
- 0.01 mbar assumes a volume of 100 m³ vapour

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.

Effects 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 sublimate. 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. Pressure rise test

The pressure rise test is a good way to confirm that there is no more vaporizing ice in the product. In the automatic cycle, parameters set on the engineering screen of the control system will be followed. It can also be performed manually by the operator. When the secondary drying stage is considered to be finished, a pressure rise test will ensure it is. The shelf temperatures and the vacuum levels will still be in secondary drying conditions. The isolation valve is closed and the operator will watch to see the rate at which the vacuum level in the product chamber increases. The vacuum level will rise even with dry product, however the rate at which it rises when ice is still vaporizing is much greater. The operator must perform tests to become familiar with these rates of pressure rise.

2.6. Air admittance

After the vials have been closed the vacuum in the chamber must be released by admitting air. Air will enter through the air admittance valve until the pressure is equalized and the door will release. This may take a few minutes.

2.7. Defrosting

Defrosting of the ice condenser is carried out manually. First the operator must release the vacuum in the condenser chamber and remove the door. A suitable collecting vessel should be positioned under the door and drip deflector. Then defrost should be pressed. The LED will light up. If it does not make sure that the condenser refrigeration is not switched on. The Ice will melt and drain out.

Defrost temperature is adjustable on the control panel. Press the down arrow once and the HI LED will light up. While this is lit scroll up or down to select a temperature. The default is 30°C.

3. Installation and Commissioning of the Unit

3.1. Site of installation

In order to ensure the air circulation of the heat exchanger, do not place items behind the unit that could restrict air flow. The ambient temperature should be between approximately +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 20cm 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.

3.2. 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 **HS-2 & HSL-2** requires a three phase power supply 415 Volts. The freeze dryer must be on a circuit protected with a 16 Amp fuse or circuit breaker.

3.3. Air admittance

Filtered air can be admitted into the product chamber. If an inert gas is needed such as nitrogen then a supply of this is required usually a gas cylinder. Take care not to over pressure the system.

3.4. Draining the condensate

The condenser door must be removed and a suitable collecting vessel positioned before defrost is activated.

3.5. 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 outside. A hose can be connected to the exhaust flange of the vacuum pump 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. Operating the HS-2 & HSL-2

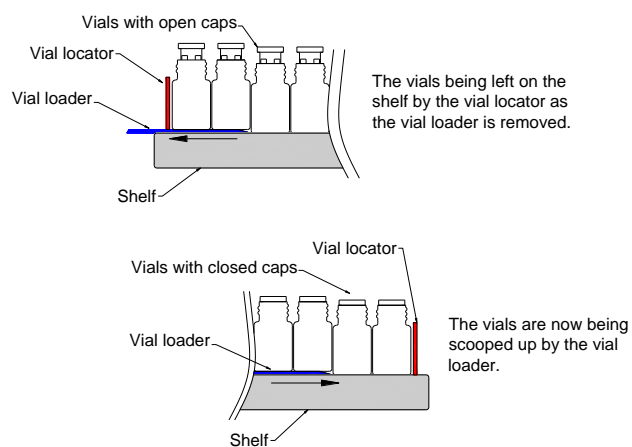
4.1 Loading Trays



4.1.1. Vial trays

The vial trays are bottomless to allow direct thermal contact between vial and shelf. The vials are loaded onto the shelf with the use of a vial loader. The vial loader is then slid out and all the vials are kept in place within the vial locator.

The vial loader slides from underneath the vials. When the product is to be unloaded the vial loader is slid back under the vials so they are supported. The vials, vial locator and vial loader can be removed as one.



There are 6 usable shelves and each shelf holds 2 shelf locators. The vial locators can hold various vial diameters. Shelf spacers can be removed and replaced with the correct size to suit any vial height.

4.1.2. Bulk trays

Bulk trays can simply slide onto the shelves. Trays can be supplied in various sizes to suit the application.

4.2. Vacuum Pump

The vacuum pump fitted to the HS-2 & HSL-2 has a 200l/min displacement.

The pump can be activated by the control panel. It also has a rocker switch on the motor which isolates the pump from the freeze drier. This should always be on for normal operation.

There is an interlock that prevent the vacuum pump from starting in ways that could cause problems.

The condenser must be below -30°C before the vacuum pump will start.

This can be overridden by pressing and holding the vacuum pump button down for a few seconds until you here a beeping sound. This override is not intended for normal operation, just testing purposes.



The vacuum pump oil should be changed when the pump is warm. The oil level should be visible in the sight glass. The intervals for changing the oil will depend upon the products being freeze dried. With the -80°C condenser chamber version it will offer better pump protection than the -55°C The condenser chamber temperature must be lower than the temperature corresponding to the vapour pressure that the product is going to freeze dried at. In the case of water vapour the following is a good guide:

- 1.030 mbar correspond to -30°C
- 0.370 mbar correspond to -40°C
- 0.120 mbar correspond to -50°C
- 0.040 mbar correspond to -60°C
- 0.01 mbar correspond to -70°C
- 0.003 mbar correspond to -80°C

If the product contains large quantities of chemicals that are more volatile than water then lower temperatures in relation to vacuum levels are required.

An oil mist filter should be fitted to the exhaust port of the vacuum pump.

For extra information on the pump please see the separate operating manual.

4.3. Isolation valve

The Isolation valve is manual and is to the left hand side of the condenser chamber door.

4.4. Air/inert gas admittance valve

Either filtered air or an inert gas can be admitted into the system. This is located on the inlet to the vacuum pump.

4.5. Condenser draining

The condenser can be drained out of the condenser door into a suitable container. Before pressing defrost the door should be removed so there is free drainage as the ice melts.

4.6. Circuit breakers

The control box has circuit breakers that may trip out in the event of an electrical problem. If a function is not working, this is the first place to check. There are circuit breakers for the following items. Left to right

- Refrigeration compressor 1 (Condenser chamber)
- Refrigeration compressor 2 (Specimen chamber)
- Vacuum pump
- Heater

Please see wiring diagrams for more detailed information



5. The control system

The control system for the freeze drier enables the operator to run the freeze drier fully automatically or manually. All data from the drying processes can be logged, saved and printed. The automatic cycles are easily programmed by the operator and can be saved or edited for later use. Programs can be changed while the cycle is running. There is a manual screen for complete manual control. This is often used when trying out a new product before a program is confirmed. The data logging and programming are done on the PC. The running of the program is looked after completely by the PLC. If there is a power failure the computer will safely shut down with the use of a built in uninterruptable power supply.

5.1. Manual Control screen

This screen is for manual control. To change to another screen there are selection buttons the down the left hand side.

- Overview
- Manual
- Engineering
- Recipe
- Graph

Next are the function buttons that are used to operate the freeze drier. To the right of each button is the function indicator.

Pink means that the function is off but can be activated.

Green means that the function is active.

Red means that the function is off and cannot be activated due to a current lock on the function. During an automatic run all the manual function buttons are locked.

The function buttons include.

- Shelf Cooling
- Condenser Cooling
- Vacuum Pump
- Drain Valve
- Defrost
- Isolation Valve
- Shelf heating
- Chamber Air inlet
- Ram Up
- Ram Down

Below are the temperature readouts of the shelves or chamber, probes and condenser. There are also vacuum levels in millibars of each chamber.

Set points are required to control temperatures and vacuum levels. If the product is to be lowered in temperature then it will be cooled by the shelf/chamber. There is a thermal lag between the chamber and the product due to the speed of thermal conduction. For fast cooling set the temperature of the chamber lower than the target temperature of the product probe. Once the product probe temperature has reached its set point or the chamber temperature reaches its set point then the cooling will stop. For example;

The chamber temperature is set at -60°C and the probe temperature is set at -40°C.

If the chamber reaches -60°C before the product probe reaches -40°C then the shelf/chamber will hold at -60°C until the product probe reaches -40°C.

If the probe temperature reaches -40°C before the chamber temperature reaches -60°C the product probe will control the shelf temperature.

The same principle applies when heating. The chamber can be set higher to allow the product to heat up quicker.

A further control on chamber heating is vacuum level. If vacuum pressure rises above its set point, heating is deactivated. As sublimation slows down the vacuum level will drop below the set point and heating switches back on.

The screenshot shows the 'Manual' control interface. On the left is a navigation menu with buttons for Overview, Manual, Engineering, Recipe, and Graph. The main area contains several function buttons with status indicators: Shelf Cooling (On, green), Condenser Cooling (Off, pink), Vacuum Pump (Disable, red), Drain Valve (Off, pink), Defrost (Off, pink), Isolation Valve (Off, pink), Shelf Heating (Off, pink), and Chamber Air Inlet (Off, pink). Below these are set point fields for Probe A and B, and a Heater/Vacuum Pressure Control field. At the bottom, there are two data tables. The first table shows temperatures for Probes 1-4, Shelves 1-4, and Condenser/Heat Exchanger/Con Return/Shelf Return. The second table shows vacuum levels for Chamber, R507 Mid/High, and R23 Low/High. On the right side of the data tables are control buttons: Start, Stop, Reset, Auto In Progress, Auto Running, Program Step, and Auto Complete. A 'Not Running' indicator is also present.

Probe 1	Temp °C	Shelf 1	Temp °C	Condenser	Temp °C	Condenser	Vac mBar
Probe 1	13.5 °C	Shelf 1	13.9 °C	Condenser	10.8 °C	Condenser	1,000.0000
Probe 2	13.1 °C	Shelf 2	13.6 °C	Heat Exc	-11.5 °C	Chamber	1,000.0000
Probe 3	13.6 °C	Shelf 3	13.0 °C	Con Return	11.7 °C		
Probe 4	13.6 °C	Shelf 4	13.1 °C	Shelf Return	14.6 °C		
Average	13.5 °C	Average	13.4 °C				

	Ref Bar
R507 Mid	3.33
R507 High	10.87
R23 Low	0.68
R23 High	14.56

5.2. Overview screen

Display

The screenshot displays the 'Overview' screen of a freeze drier control system. It features a central schematic diagram of the freeze drier chamber with shelves and various ports. To the left is a vertical navigation menu with buttons for Overview, Manual, Engineering, Recipe, Graph, Display, Login, Change Pwd, Minimise, and Shutdown. To the right is a control panel with buttons for Intial Vac Test, Pressure Rise Test, Use Dry Storage, Use Shelf Stoppering, Per-Stoppering Air Inlet, and Post-Stoppering Air Inlet. Below the schematic is a table of machine components. At the bottom, there are several data tables for temperature, vacuum, and pressure, along with a status control panel.

Compressor 1 R23	Isolation Valve	Compressor 2 R507	Ram Up
Condenser Solenoid	Vacuum Pump	Circulation Pump	R507 Pump Down Solenoid
Product Solenoid	Product Chamber Air Inlet	R23 Release Solenoid	Heater
Condenser Drain	Ram Down	Product Chamber Bleed	Defrost

File Name	4x Rotrol-2.25
Target	Shelf 1
Temperature	-50.0 °C
Actual	13.8 °C
Vacuum	1,000.0000 mBar
Actual	1,000.0000 mBar
Trigger Value	Temp: Probe 1 45.0 °C
Actual	13.5 °C

Probe	Temp °C	Shelf	Temp °C	Condenser	Temp °C	Vac mBar
Probe 1	13.5 °C	Shelf 1	13.8 °C	Condenser	10.8 °C	1,000.0000
Probe 2	13.1 °C	Shelf 2	13.6 °C	Heat Exc	-11.4 °C	1,000.0000
Probe 3	13.6 °C	Shelf 3	13.0 °C	Con Return	11.7 °C	
Probe 4	13.6 °C	Shelf 4	13.1 °C	Shelf Return	14.6 °C	
Average	13.5 °C	Average	13.4 °C			

	Ref Bar
R507 Mid	3.30
R507 High	10.93
R23 Low	0.58
R23 High	14.46

Start Auto In Progress
 Stop Auto Running
 Reset Program Step
 Not Running Auto Complete

Connected To 192.168.1.150 PLC Scans: 197681 SQL Scans: 309 SQL: Ok

This screen is for an overview of the freeze drier it shows what parts of the freeze drier are running. Vacuum and temperature set points, program step number and running status are also displayed. The diagram will change depending on what is in operation. In the current view the shelves are red because they are heating. The condenser is blue because it is cooling. The isolation valve between the two chambers is open. Below the picture are black boxes indicating the components of the machine operation. When they are green then that component is active and black indicates that it is inactive.

Login

The freeze drier control system has 3 levels of login access. These can be increased upon the customer's request.

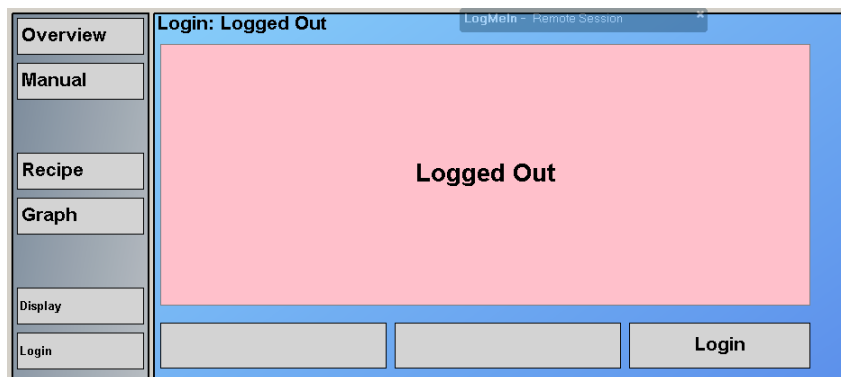
The first level has no access password, it enables the freeze drier operation in manual mode, to load a program/recipe from the list and to start/stop the automated cycle. The user can look through the screens, add and remove lines from the graph and change trace colours.

The second level also allows the programming of recipes, saving and editing. A password is required to access this level

The third level will also give the operator/engineer access to calibration settings and engineering values. A further password is required to access this level.

Whenever a function is not available due to current access level then selection buttons will not be visible.

To log in enter the login name in the first grey box and the login password in the second grey box and then press Login.



To log out click on the large green rectangle



5.3. Recipe screen

The screenshot shows the Recipe screen interface. On the left is a sidebar with buttons: Overview, Manual, Engineering, Recipe, Graph, Program, Options, Load / Save, Recv From PLC, Send To PLC, Clear, Print, Minimise, and Shutdown. The main area contains a table with columns: Step, Probe, Temperature, Max Pressure, Min Pressure, Item, and Value. The table lists 18 steps with various parameters like temperature, pressure, and time delays. Below the table is a status panel with several data readouts: Probe 1-4 temperatures, Shelf 1-4 temperatures, Condenser, Heat Exc, Con Return, Shelf Return temperatures, Condenser and Chamber vacuum levels, and Ref Bar levels (R507 Mid, R507 High, R23 Low, R23 High). On the right side of the table, there are 'Up' and 'Down' buttons. At the bottom of the status panel, there are buttons for Start, Stop, and Reset, along with status indicators for Auto In Progress, Auto Running, Program Step, and Auto Complete.

The Recipe screen is for programming the automatic freeze drying cycle. To change to another screen there are selection buttons down the left side as on previous pages.

There are also options for:

- **Program** - Creating a new programs
- **Options** – Pre programmed options
- **Load/save** - Save or load a program
- **Receive from PLC** - the details of a program
- **Send to PLC** – the details of a program.
- **Clear** – Clear the programming table

Program setup

The program setup requires the input of a sequence of steps to complete a freeze drying cycle. The operator will define the running conditions within a step and an achievable value to prompt the next step. This value may be a temperature, a vacuum level or a time delay.

When a temperature is entered, the operator will need to specify which temperature probe is to be used.

On this freeze drier the shelves are not temperature controlled, only the chamber.

There are 8 mobile product probes, these are listed:

Probe 1, Probe 2.....

The following is a simplified example of programming a freeze drying cycle to explain the procedure.

Entering the first step

The Shelves are chosen as the target temperature. A triggering condition is set to initiate the next step.

To freeze the product to -40°C the operator could do the following:

The first method

Select the required item such as Chamber temperature at -50°C which would allow the chamber to cool down to this point. As the chamber cools down so will the product. The trigger item would be a product probe 1 set at -40°C. Upon reaching this the next step will be activated. Depending on the type or quantity of product, the thermal lag between the chamber may vary. In the time it takes the product to reach -40°C the chamber may or may not reach -50°C however they will not go below.

The disadvantage of this is:

Setting a minimum chamber temperature gives a constant temperature to absorb heat from the product. As the product temperature gets closer the temperature difference gets less. Therefore the rate of cooling is reduced. Freezing will take longer.

The advantages of this are:

The overall Product temperature will be much more even.

There is less chance of product temperature overshoot.

The chamber does have electrical fans that will operate when cooling if there is not a vacuum present. This will give a more equal temperature throughout the chamber.

The second method

Select the required item as a probe 1 temperature at -40°C. Set the trigger item probe 1 at -40°C. This way the chamber will cool as cold as is possible for the refrigeration system.

The disadvantages of this are:

The overall Product temperature may be much less even.

There is more chance of product temperature overshoot.

The advantage of this is:

Fastest rate of cooling due to greater temperature differences between product and shelf

(Having said this the theory behind this bespoke machine may be proven wrong in practice!!)

Entering the second step

A time step could be used to allow the product to stabilize in temperature. The temperature of the product probe can stay the same so probe 1 is set to -40°C. If the time step was set to one hour, the next step would be triggered after this time.

Entering the third step

The third step could be to create a vacuum on the product.

The temperature of the product would not need further cooling so probe one would remain at -40°C. A vacuum trigger would be selected next with a value of 0.1mbar. The condenser chamber would be automatically cooled to -30 °C and the vacuum pump would start.

Once 0.1mbar has been reached the next step would trigger.

As this is the first step that the vacuum is used a value can be put into the target vacuum box. This is the vacuum control setting. This setting will prevent the heater from activating while the vacuum level is above the value entered. As the probe 1 temperature is set at -40°C the system would try and maintain this using heating or cooling. As the vacuum level improves the product will cool by sublimation so the heater would attempt to counter this.

Entering a value into the target vacuum box the same as the trigger value would prevent the heater from activating and allow natural vacuum cooling. The temperature will drop depending upon the level of vacuum created. If the box is left empty or a higher value is entered than the value in the target box then the heater would attempt to maintain probe 1 at the set temperature.

Due to the heat input there will be more sublimation occurring while initial vacuum is being drawn. This will increase the length of time taken to achieve the initial vacuum level.

Entering the fourth step

The fourth step would be to heat the product up to 40°C while maintaining a vacuum level below a set point. This set point is of most importance at the beginning of the primary drying stage when the sublimation rate is at its highest. If the vacuum level rises above a set value then the ice in the product will start to melt. The vacuum control level will ensure that the ice core of the product stays frozen. To achieve the maximum rate of sublimation the vacuum level must correspond to a vapour pressure of a temperature as close to the eutectic point as is safely possible.

With regards to setting the temperatures similar methods as in the first step can be applied. Set a maximum shelf 1 temperature, reducing the chances of overshoot, but expect heat transfer to take longer. Or allow the shelves to heat up to their maximum temperature allowed (settings are in the engineering page) increasing the heat transfer rate, but also increasing the chances of product temperature overshoot.

Entering the fifth step

The fifth step could be a time step to allow the product to stabilize in temperature.

The temperature of the product probe 1 can stay at 40°C. If the time step was set to one hour, the next step would be triggered after this time.

Entering the sixth and final step

The sixth step in this example would be secondary drying. This can be considered as another vacuum step. The temperature of the product does not need further cooling. Select a trigger item as vacuum, with a value of 0.05mbar. When 0.05mbar is achieved it is the end of the programmable steps. If selected there may be some post steps to complete.

Notes:

Usually programming an automated freeze drying process will involve many more steps detailed steps. As there are several shelves it is sometimes important to repeat each temperature step for each shelf or product probe. This will ensure that every probe reaches the set points required.

Options

The operator may choose to activate the pre-programmed steps listed as

- **Initial Vacuum test**

This ensures that the chambers are going to be vacuum tight before the process is allowed to start. Once the program has started an initial vacuum is pulled on the chambers. As long as a slight vacuum can be reached the program will know that it is safe to start, that the door has not been left open or fittings/seals are missing.

- **Pressure rise test**

The pressure rise test if selected will occur after the last of the programmed steps has ended. The isolation valve will close. An initial vacuum reading in the product chamber will be taken. After a preset timer has lapsed another vacuum reading will be taken. If the pressure rise is within an allowable range then the next post step will activate. If the pressure rise is too high then the valve will re-open and drying will continue for a time span before retesting.

- **Dry storage**

This is a means of storing the product within the freeze drier at a controlled temperature once the process has ended. This is useful if the product finishes its process in the middle of the night and there are no operators to unload it. If products are usually transferred to a fridge after drying then the freeze drier can match these conditions for ideal storage until unloading can take place.

- **Use Shelf Stoppering**

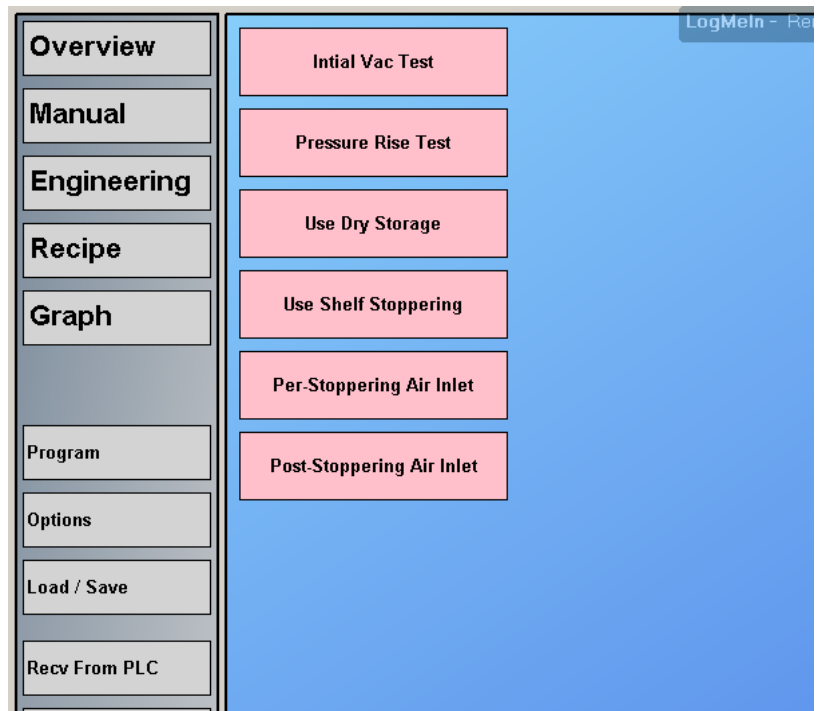
If the freeze drier is required to stopper vials automatically as soon as the product is dry, then this should be selected. At the end of the program the shelves will close together pushing the stoppers into the vials.

- **Pre – Stoppering Air Inlet**

It may advantageous to fill the vials with a charge of inert gas before stoppering. Perhaps, nitrogen or argon, this may be at a partial vacuum level. By selecting this option the chamber can be raised in pressure via the vacuum bleed valve using a chosen gas before vial stoppering takes place.

- **Post – Stoppering Air Inlet**

This option will allow the chamber pressure to rise only after vial stoppering. This will ensure that the vials are closed with a vacuum inside.



Load / Save

This will open up File Manager. The operator can load a saved Recipe off the list or save one that is currently programmed.

Receive from PLC

The PLC contains the recipe that the freeze drier will operate with. If the program list has been cleared or programming mistakes have been made then this button will re-populate the programming list with current values on the PLC.

Send to PLC

Once a program has been altered, created or loaded then it must be sent to the PLC. This will enable the PLC to operate the freeze drier with the up dated program rather than the last programming it was using.

Clear

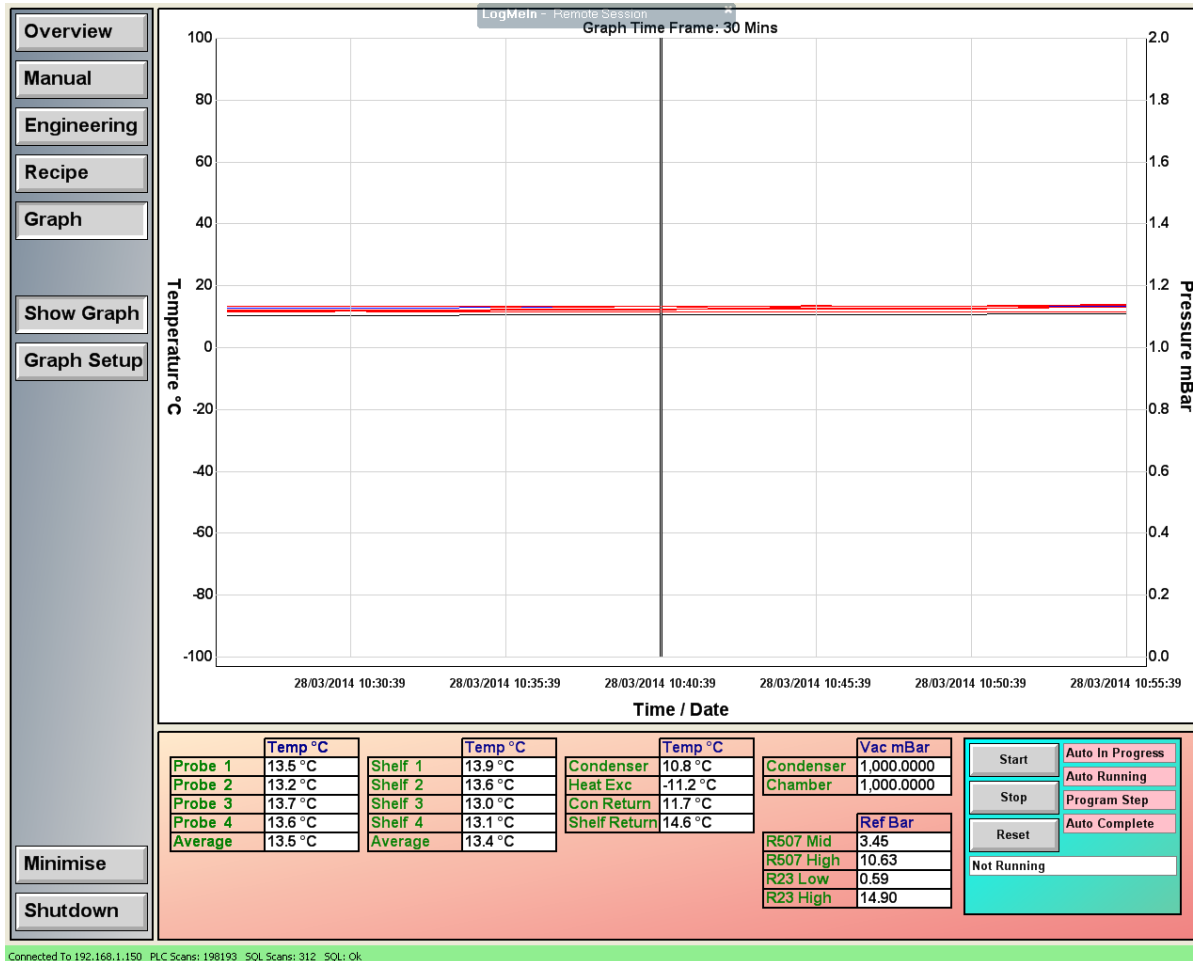
To delete all program step data off the list and return to a clean unpopulated list press Clear.

The screenshot shows the 'File Manager' window with a sidebar on the left containing buttons for Overview, Manual, Engineering, Recipe, Graph, Program, Options, Load / Save, Recv From PLC, Send To PLC, and Clear. The main area displays a list of programs: Jam, PROGRAM, PROGRAM2, Recipe, TEST, TESTRUN, and TRIAL 1. There are 'Up' and 'Down' buttons on the right side of the list. At the bottom, there are 'Load Recipe' and 'Save Recipe' buttons.

The screenshot shows the main interface with a sidebar on the left containing buttons for Overview, Manual, Engineering, Recipe, Graph, Program, Options, Load / Save, Recv From PLC, Send To PLC, and Clear. The main area displays a table with the following data:

Step	Probe	Maintain			Trigger	
		Temperature	Max Pressure	Min Pressure	Item	Value
1	Shelf	-35.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	-35.0 °C
2	Shelf	-35.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	60 m
3	Shelf	-35.0 °C	1,000.0000 mBar	0.0000 mBar	Vac	0.1000 mBar
4	Shelf	-30.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	-30.0 °C
5	Shelf	-30.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	28 m
6	Shelf	-20.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	-20.0 °C
7	Shelf	-20.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	333 m
8	Shelf	-4.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	-4.0 °C
9	Shelf	-4.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	135 m
10	Shelf	20.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	20.0 °C
11	Shelf	20.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	120 m
12	Shelf	4.0 °C	1,000.0000 mBar	0.0000 mBar	Temp: Shelf	4.0 °C
13	Shelf	4.0 °C	1,000.0000 mBar	0.0000 mBar	Time Delay	60 m
14	Probe Avg	0.0 °C	1,000.0000 mBar	0.0000 mBar	Select Trigger	Select Trigger

5.4. Graph



Show Graph

The data logging monitors temperatures of all mobile probes, shelves, condenser and vacuum levels. Information on temperature and vacuum changes becomes very easy to analyze on a line graph.

Graph Setup

The screenshot shows the 'Graph Setup' window with a sidebar on the left containing navigation buttons: Overview, Manual, Engineering, Recipe, Graph, Show Graph, Graph Setup, Minimise, and Shutdown. The main area is titled 'Graph Setup' and includes a '360 Mins' dropdown menu. Below this are two columns of sensor settings:

- Thermo-couple:** A list of sensors (Probe Avg, Probe 1-4, Shelf Avg, Shelf 1-4, Condenser, Heat Exc, Condensor Return, Shelf Return) with color-coded boxes and 'On/Off' toggle buttons.
- Pressure Unit:** A list of pressure points (Condenser, Chamber, R507 Mid Stage, R507 High Stage, R23 Low Stage, R23 High Stage) with color-coded boxes and 'On/Off' toggle buttons.

At the bottom of the window, there are three data tables and a control panel:

	Temp °C		Temp °C		Temp °C
Probe 1	13.5 °C	Shelf 1	13.9 °C	Condenser	10.8 °C
Probe 2	13.2 °C	Shelf 2	13.7 °C	Heat Exc	-11.3 °C
Probe 3	13.7 °C	Shelf 3	13.0 °C	Con Return	11.7 °C
Probe 4	13.6 °C	Shelf 4	13.1 °C	Shelf Return	14.6 °C
Average	13.5 °C	Average	13.4 °C		

	Vac mBar
Condenser	1,000.0000
Chamber	1,000.0000

	Ref Bar
R507 Mid	3.43
R507 High	10.69
R23 Low	0.59
R23 High	14.80

The control panel on the right includes buttons for Start, Stop, and Reset, along with status indicators for Auto In Progress, Auto Running, Program Step, Auto Complete, and Not Running.

At the very bottom, a status bar reads: 'Connected To 192.168.1.150 PLC Scans: 198128 SQL Scans: 312 SQL: Ok'

Within Graph setup

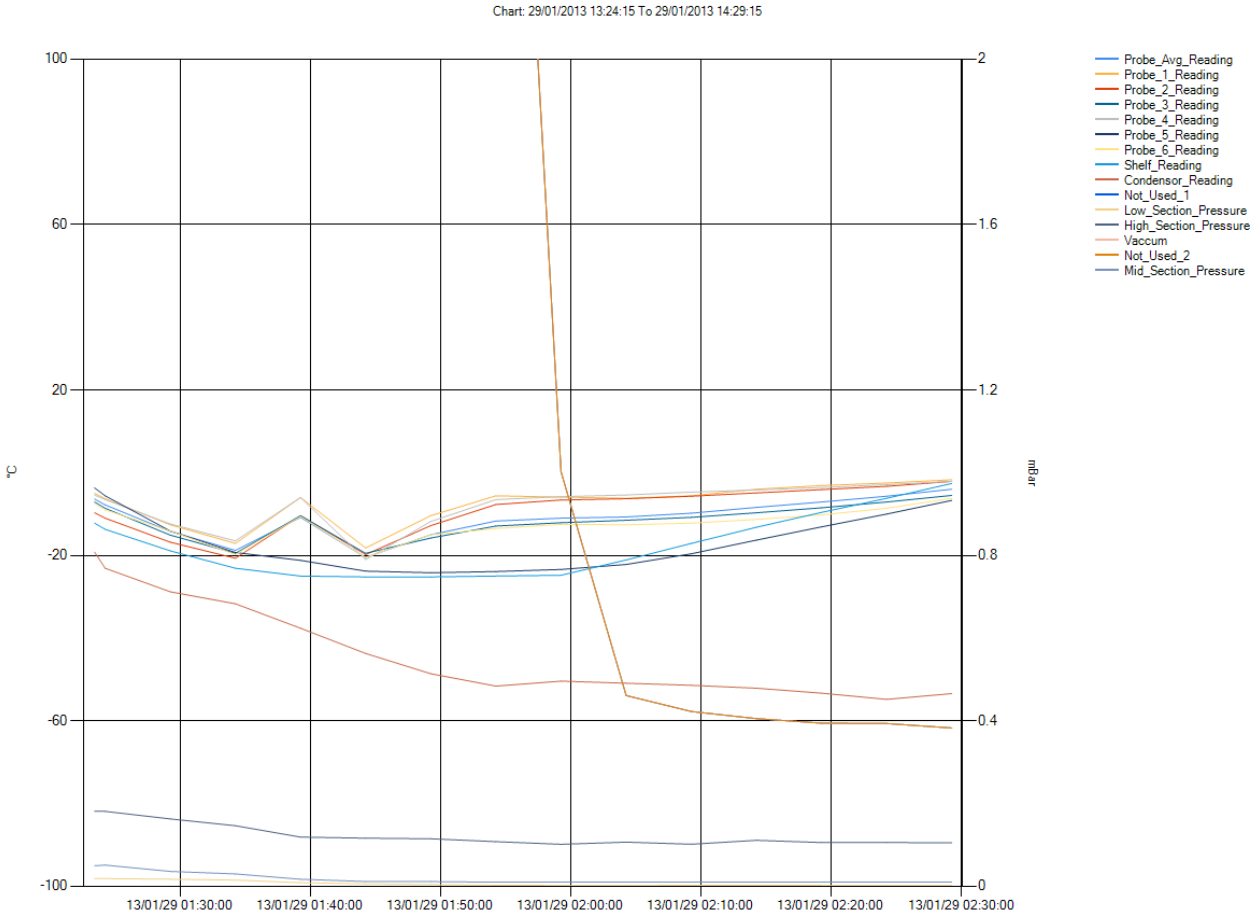
The time scale that the graph shows can be selected, 30mins to 96 hours. Blue will indicate the time scale that has been selected.

Thermocouple can be identified on the graph by changing the line colour. They can also be hidden by turning them off.

Vacuum pressures can be identified on the graph by changing the line colour. They can also be hidden by turning them off

The Graph print out

Below is an example of a cycle printout. On the left there is a key to identify graph lines. Product and Batch identification appear as the heading.



5.5. Engineering

The Engineering screen is for programming the automatic freeze drying cycle. To change to another screen there are selection buttons down the left side as on previous pages.

There are also options for:

- **Parameters** – Engineering parameters
- **TC Calibration** – Temperature Calibration
- **Pressure Calibration** – Pressure Calibration
- **Configuration** - configuration

Engineering Parameters

Time allowed For Initial Vacuum Test

This is the time that the vacuum pump will run during an initial vacuum test to confirm the chamber is leak tight before an automated program can start. A moderate vacuum level is set of approximately 600 mbar to confirm doors are closed and the vacuum pump is switched on at the motor. Depending on the volume of the chamber, a sensible time is allowed for this test. As soon as the vacuum level is achieved within the time frame, the test is complete. If the required vacuum is not achieved within the set time the program will not commence. The problem will require investigation.

Initial Vacuum Test Pressure

This is the box were the vacuum level is entered. As mentioned above a sensible setting is 600 mbar, this is only achievable with a leak tight vacuum system. This vacuum level will not adversely affect unfrozen product.

Max Shelf / Chamber Temperature

A value can be set so that if any temperature goes above it all heating will stop and a warning will be displayed. This is regardless of which sensors are used for temperature control. It is an overall safety limit incase the thermocouple used for temperature control acquires a fault or is poorly programmed or positioned.

Max Probe Temperature

A value can be set so that if any temperature goes above it all heating will stop and a warning will be displayed. This is regardless of which sensors are used for temperature control. It is an overall safety limit incase the thermocouple used for temperature control acquires a fault or is poorly programmed or positioned.

Vacuum stability Time

The vacuum readings that initiate a change in programmed steps must remain beyond the set point threshold for this selected time. This will ignore sporadic readings on while on the edge of the vacuum sensors designed operating range.

Vacuum start temperature

This is the temperature set point that does not allow the vacuum pump to start until the condenser temperature is below it. Usually set at -30°C. This will stop unfrozen water vapour being drawn into the vacuum pump.

Dry product storage probe

The screenshot shows the 'Engineering Parameters' screen. On the left is a vertical menu with buttons for 'Overview', 'Manual', 'Engineering' (highlighted), 'Recipe', 'Graph', 'Parameters', 'TC Calibration', 'Pressure Calibration', and 'Configuration'. The main area on the right contains a list of parameters, each with a text input field and a label:

Parameter Value	Parameter Label
3.00 mins	Time Allowed For Initial Vacuum Test
600.0000 mBar	Initial Vacuum Test Pressure
50.0 °C	Max Shelf / Chamber Temperature
50.0 °C	Max Probe Temperature
0.20 mins	Vacuum Stability Time
-30.0 °C	Vacuum Start Temperature
Shelf	Dry Storage Probe
4.0 °C	Dry Storage Temperature
30.00 mins	Defrost Time Period
40.0 °C	Maximum Defrost Temperature
1.00 mins	Refrigeration Turn Off Delay
1.00 mins	Time Allowed For Pressure Rise Test
0.1000 mBar	Pressure Test Rise Allowed
3.00 mins	Time Before Pressure Rise Re-Test
10.0 s	Stoppering Time
300.0000 mBar	Pre-Stoppering Air Inlet Pressure

This is the product or shelf probe that is used to control the temperature when the freeze dryer is in dry storage mode. Dry storage can be selected to run after the automated freeze drying cycle has ended if the product is not unloaded straight away. If a cycle ends during the night and the operator cannot unload the freeze drier, preferred conditions such as fridge temperatures can be matched on the shelves. A product or shelf probe may be chosen for this control.

Dry product storage temperature

This is the temperature that dry product will be controlled to if dry product storage is selected.

Defrost time

This is the time that the condenser chamber will be heated for. A setting that will give enough time to melt all the condensed ice is required. This can be set by the operator to suit the application of the freeze drier.

Maximum defrost temperature

This is the maximum temperature that the condenser temperature is allowed to reach while defrosting.

Refrigeration turn off delay

The compressors of the refrigeration system will continue to run for a period of time after the refrigeration has been switched off. This will prevent liquid refrigerant being left in areas of pipe work that are exposed to high temperatures.

Time allowed for pressure rise test

The end of the automated cycle has the option a pressure rise test. This is where the product is sealed off in the product chamber to see if sublimation has finished. If the pressure rises above the set limit (pressure rise quantity) it can be considered to not be dry, as sublimation is still occurring. If the pressure rise is within limits the product is dry. For the pressure rise test there is a set time frame that the pressure rise can occur within.

Pressure rise allowed

The end of the automated cycle has the option a pressure rise test. This is where the product is sealed off in the product chamber to see if sublimation has finished. If the pressure rises above the set limit it can be considered to not be dry as sublimation is still occurring. If the pressure rise is within limits the product is dry. For the pressure rise test there is a set vacuum level that is allowable. A rise above this pressure indicates that the product is not dry and the test has failed.

Time before pressure rise retest

A fail is if the pressure rises beyond the allowable **pressure rise quantity** within the **Time allowed for pressure rise test**. Time before retest is the time that the product will have back under drying conditions before the next test.

Stoppering time

During automatic vial closing the ram will raise up to close the shelves together and seal the vials. This is the time allowed for this procedure and is set at the factory.

Pre – Stoppering air inlet pressure

If a pressure of inert gas is required to fill the vials before they are sealed then the level of vacuum can be entered.

Thermocouple Calibration

Overview

Manual

Engineering

Recipe

Graph

Parameters

TC Calibration

Pressure Calibration

Configuration

Minimise

Shutdown

LogMeIn - Remote Session

Temperature Calibration

Thermo-couple	Status	Temperature	Point B Reading	Point B Desired	Offset	Graph	
Probe Avg	Not Used	3.4 °C	0.0 °C	0.0 °C	0.0 °C	Off	On
Probe 1	Ok	4.8 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Probe 2	Ok	4.0 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Probe 3	Ok	3.2 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Probe 4	Ok	4.9 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Probe 5	Ok	0.8 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Probe 6	Ok	2.7 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Shelf	Ok	-1.7 °C	0.1 °C	0.1 °C	0.0 °C	On	On
Condensor	Ok	-8.2 °C	0.1 °C	0.1 °C	0.0 °C	On	On

Temp °C	
Probe 1	4.8 °C
Probe 2	4.0 °C
Probe 3	3.2 °C
Probe 4	4.9 °C
Probe 5	0.8 °C
Probe 6	2.7 °C
Average	3.4 °C

Temp °C	
Condenser	-8.2 °C
Shelf	-1.7 °C

Vac mBar	
Vacuum	22.6986
LP	3.380
MP	2.880
HP	3.400

Start Auto In Progress

Stop Auto Running

Reset Program Step

Auto Complete

Not Running

Connected To 192.168.1.150 PLC Scans: 138 SQL Scans: 1 SQL: Ok

This screen is for factory setup or periodic temperature calibration. Sensor connection status, PLC temperature reading, correction scaling and offset can be set to give the correct reading.

Vacuum / Pressure Calibration

Overview

Manual

Engineering

Recipe

Graph

Parameters

TC Calibration

Pressure Calibration

Configuration

Minimise

Shutdown

Absolute Pressure Calibration (Vacuum Calibration)

Pressure Unit	Point A mV	Point A Reading	Point B mV	Point B Reading	Reading	Colour	
Condenser	1000	400	9000	6800	1,185.7684 mBar		Off
Chamber	1000	400	9000	6800	1,177.6062 mBar		On
Low Section	0	0	800	8000	3.24 Bar		On
High Section	100	0	3100	8000	9.83 Bar		On

	Temp °C		Temp °C		Vac mBar
Probe 1	15.4 °C	Condenser	12.5 °C	Condenser	1,000.0000
Probe 2	15.2 °C	Shelf	14.6 °C	Chamber	1,000.0000
Probe 3	15.4 °C	Heat Exc	15.0 °C		
Probe 4	15.4 °C				
Probe 5	15.1 °C				
Probe 6	14.7 °C				
Average	15.2 °C				

	Ref Bar
R607 LP	3.24
R607 HP	9.83

Start	Auto In Progress
Stop	Auto Running
Reset	Program Step
	Auto Complete
Not Running	

Connected To 192.168.1.150 PLC Scans: 1213029 SQL Scans: 1132 SQL: OK

This screen is for factory setup or periodic vacuum calibration. Sensor connection status, PLC Vacuum reading, correction scaling and offset can be set to give the correct reading.

Operating Manual Freeze Drier HS-2 & HSL-2

Page 34

Engineering Configuration

Overview

Manual

Engineering

Recipe

Graph

Parameters

TC Calibration

Pressure Calibration

Configuration

Minimise

Shutdown

Engineering Configuration LogMeIn - Remote Session

Off: Diagnostic Mode (Recommend Off)

On: Advanced Mode (Display Extra Data)

Off: Office Mode (Recommend Off)

Off: No Login (Recommend Off)

	Temp °C
Probe 1	15.4 °C
Probe 2	15.1 °C
Probe 3	15.4 °C
Probe 4	15.4 °C
Probe 5	15.1 °C
Probe 6	14.7 °C
Average	15.2 °C

	Temp °C
Condenser	12.5 °C
Shelf	14.7 °C
Heat Exc	15.0 °C

	Vac mBar
Condenser	1,000.0000
Chamber	1,000.0000

	Ref Bar
R507 LP	3.26
R507 HP	9.82

Start Auto In Progress

Stop Auto Running

Reset Program Step

Auto Complete

Not Running

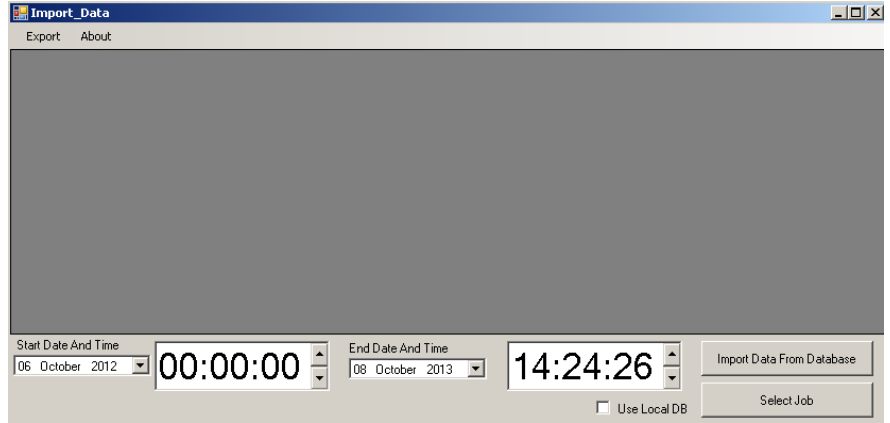
Connected To 192.168.1.150 PLC Scans: 1213230 SQL Scans: 1132 SQL: Ok

Several configurations can be selected, this is for online support of the freeze drier or the software. Extra information can be displayed that would not be needed for the everyday operator. There are several levels/layers of information that displayed. This is only accessible with the higher level of login access.

6. Reporting

There is a continual data logging process taking place whenever the freeze drier operating system is in operation. The data logging application is accessible in the windows desktop. Minimize the freeze drier application and open “Reporting”.

- 1) Enter the start date and time of the data you wish to view.
- 2) Enter the end date and time.
- 3) If unchecked select “Use Local DB”
- 4) Press “Import Data From Database”



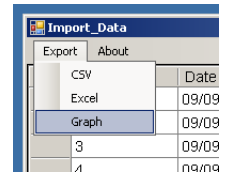
The screenshot shows the 'Import_Data' application window with a data table. The table has the following columns: Index_ID, Date, Comms, Program_Count, Step_Number, Out_IO, Probe_Avg_St, and Probe_Av. The data is as follows:

Index_ID	Date	Comms	Program_Count	Step_Number	Out_IO	Probe_Avg_St	Probe_Av
1	09/09/2013 ...	Connected ...	0	0	0	Not Used	3163.6
2	09/09/2013 ...	Connected ...	0	0	0	Not Used	3163.6
3	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.7
4	09/09/2013 ...	C(Connected To 192.168.1.150 PLC Scans: 196 SQL Scans: 1				Not Used	3160.8
5	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8
6	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8
7	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8
8	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8
9	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8
10	09/09/2013 ...	Connected ...	0	0	0	Not Used	3160.8

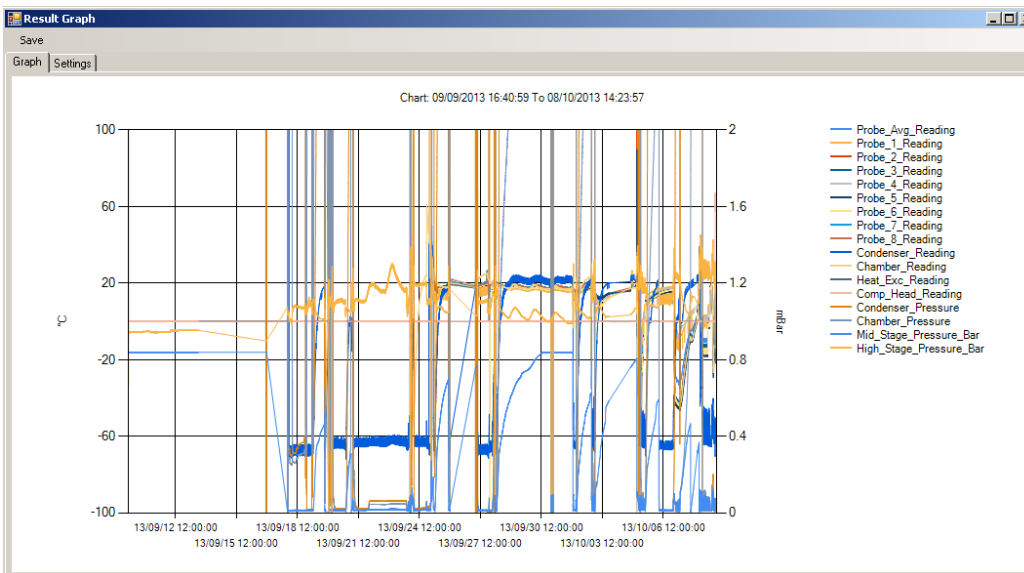
At the bottom of the window, there are date and time selection fields set to '06 October 2012' and '08 October 2013' with times '00:00:00' and '14:24:26'. There is a checkbox labeled 'Use Local DB' which is checked, and a 'Select Job' button.

The data base can be scrolled through to find out temperatures vacuum levels and refrigeration pressures for every data logging interval. Each data logging interval is currently set at 1 minute. An extension of this data will show every active function and PLC output.

Select Export send data to CSV, Excel or Graph file.



A graph can be generated using the data selected. Trace colours can be changed or removed in the settings page to show the information that important.



7. Troubleshooting

7.1. Power failure

The freeze drier will resume its program or settings 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 in which 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, it is recommended that the product is refrozen.

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

If the product is considered to be still usable then freeze drying can restart.

7.2. Insufficient vacuum

Is the vacuum pump is running? If not:

- Check that the pump motor switch is on
- Check that the IEC connection lead is connected
- Check that the vacuum pump circuit breaker has not switched off.
- Check that the contactor is operating.

If the vacuum is sufficient on the condenser chamber but significantly worse on the specimen chamber:

- Check that air admittance and inert gas valves are closed.
- Check all vacuum connections on specimen chamber including door seal. Repair or replace.
- Check for oil leakages from the shelf thermal fluid. Oily residue may collect in the chamber.
- Check for water from cleaning or spilled product residue, this may vaporize preventing vacuum.
- Remove panels and listen for leaks after a vacuum evacuation has been tried.

If the vacuum cannot be achieved in the condenser chamber with the isolation valve closed:

- Check all vacuum connections on condenser chamber including door seal. Repair or replace.
- Check the condition of the isolation valve sealing surface. Glass pieces from broken vials can slit the rubber seal. If there is a doubt about this coat in vacuum pump oil or vacuum grease and test to see if there has been an improvement in the vacuum level.
- Check that there is no suction on the air inlet. This would indicate a leaking valve.
- Check the vacuum pump is pulling an adequate vacuum, test with a separate vacuum sensor.
- Check that the isolation valve is sealing by opening it and pulling a vacuum on the product chamber as well, eliminating the seal of the valve.

If the vacuum pump cannot achieve a sufficient vacuum then replace it or get it repaired.

7.3. Insufficient cooling of the condenser chamber

If the condenser chamber is not reaching the relevant temperature -55°C or -85°C

(Please allow 15% for high load conditions)

- Check to see if there is liquid flow in the refrigeration sight glass. Contact the manufacturer, supplier or a local refrigeration engineer.
- Check the circuit breakers for compressor 1, compressor 2 and fans.
- Check that the contactors are operating.
- Check the overview screen to ensure that refrigeration and condenser cooling are operating.

7.4. Insufficient defrosting of the condenser chamber

If defrost is not heating up.

- Check the circuit breaker for defrost
- Check that the contactor is operating.
- Ensure that defrost LED is activated.
- Check that temperature control is not controlled by a sensor that is malfunctioning and reading an incorrectly high temperature.

7.5. Insufficient cooling of the shelves

If the shelves are not reaching low set points or relevant temperature -45°C or -60°C

- Check to see if there is liquid flow in the refrigeration sight glass. Contact the manufacturer, supplier or a local refrigeration engineer
- Check the circuit breakers for compressor 1, compressor 2, fans and circulation pump
- Check that all the contactors are operating.
- Check the overview screen to ensure that refrigeration, shelf cooling and circulation pump are operating.
- Check that temperature control is not controlled by a sensor that is malfunctioning and reading an incorrectly low temperature.

7.6. Insufficient heating of the shelves

If the shelves are not reaching high set points or the relevant temperatures 60°C to 80°C.

- Check the circuit breaker for heater and circulation pump.
- Check that all the contactors are operating.
- Ensure that heater and circulation pump are activated.
- Check that shelf temperature control is not controlled by a sensor that is malfunctioning and reading an incorrectly high temperature.

7.7. Doors will not seal under vacuum

If the doors will not seal under vacuum

- Check that the vacuum pump is running
- Close the isolation valve and check that the condenser door is sealing
- Check that the air admittance valve is closed.
- Clamp the product chamber door and check that it is pushed up against its seal all around.
- Open the isolation valve and release the door clamps.

Does the door still open?

7.8. Incorrect probe temperature

Incorrect probe temperatures can be due to

- Unplugged probes
- Broken thermocouples
- Thermocouples with loose connections

7.9. Trays are distorting

Large trays can have a tendency to distort with high and low temperature. Avoid using large trays where possible. When they are cold they tend to twist and when they are warm they tend to bow. The shelf to tray contact becomes less and freeze drying rates are reduced.

8. Maintenance

8.1. Condenser chamber

Before each start-up, ensure that condenser chamber is drained.

8.2. Heat exchanger

The refrigeration heat exchanger is used to cool the refrigerant compressed by the refrigeration unit. The heat exchangers are located on each side of the unit and should be kept free of dust or dirt residues. The heat exchanger can be cleaned by brushing, 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 seals

Special attention must be paid to the rubber seals. If the seals are dirty, they must be removed, cleaned and slightly greased with vacuum grease. If they show signs of cracking they must be replaced.

8.4. Vacuum pump

Clean up any oil spills on or around the freeze drier from oil changes.

For the maintenance of the vacuum pump, please refer to the separate operating manual.

Additionally:

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

The exhaust filter should be replaced if it becomes saturated. Where possible there should still be a pipe after the exhaust filter leading into open air. Take care that the condensate in the filter does not rise too high.

8.6. Cleaning 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, solvents or agents with abrasive particles. Remove product residues from the ice condenser chamber using a cloth. It is recommended to open the doors of the freeze drier when it 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.

8.7. Checks by the operator

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

- Doors and hinges
- Seals
- Oil level of vacuum pump
- Heat exchangers
- Exhaust filter

9. IQ/OQ Qualification

This Qualification Protocol is solely intended to be used with HSL-2 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 HSL-2 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 downloaded 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 Check in the 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 an IEC connection. (An oil mist eliminator exhaust filter is recommended.)	Y	N
1d	Accessories	The HS-2/HSL-2 is not the type of Freeze dryer that requires accessories for it to have freeze drying function. Variations in Tray design will allow freeze drying of different products. Have the correct trays been chosen for the product?	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 doors/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	Door Seal Installation	Is the door seal installed? Does the door seal against it when closed? Does it make contact all around the chamber?	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 on the condenser chamber with the correct rubber hose and clamps provided?	Y	N
2e	Vacuum Pump Electrical	Is the vacuum pump plugged into 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	Condenser Chamber Refrigeration	With the freeze dryer system at ambient temperature, turn the Main Power Switch Switch on Condenser Chamber Cooling Does the refrigeration system start? Record the time it started. _____	Y	N
1b	Power to Vacuum Pump	Ensure the condenser temperature has gone below the vacuum start temperature. See engineering settings screen. Press the button labeled "Vacuum Pump." Does the vacuum pump start? Record the time it started. _____	Y	N
1c	Condenser Refrigeration Effectiveness	Does the condenser chamber temperature reach -35°C in less than 50 (With the system under vacuum and 21°C ambient temperature.) NOTE: Freeze Dryers are tested to -55°C or -85°C in the factory. Conditions may vary in the field resulting in warmer acceptance temperatures.	Y	N
1d	Shelf Refrigeration	Set the Shelf Cooling to -45°C Does the refrigeration system start? Record the time it started. _____		
1e	Shelf Refrigeration Effectiveness	Does the Shelf temperature reach -35°C in less than 50minutes for the NOTE: Freeze dryers shelves are tested to -40°C or -70°C in the factory. Conditions may vary in the field resulting in warmer acceptance temperatures.	Y	N
1f	Verify Displayed Temperature	<u>Condenser temperature</u> indicated on the display is measured on the wall of the horizontal condenser chamber. The wall of the chamber can vary in temperature at various points front, back top, bottom and sides. Take several readings. The average should be within 5°C of the readout. Is at least one position the same as the display or lower? Ref. Instrument? _____ If the temperature does not correlate, contact Frozen in Time 01347 878158	Y N/A	N

1h	<p><u>Shelf temperature</u> indicated on the display is measured in the fluid inside the shelf. The shelf contains fluid that is circulating. As long as the circulation pump is running and there is no trapped air the temperature of the shelves should be very consistent.</p> <p>Set the Shelf temperature at various points Take readings on every working shelf. This may take several stages depending on how many mobile probes are fit.</p> <p>Attach the probes using suitable adhesive tape and apply the tape when the shelves are dry and at approximately room temperature. If the tape becomes detached during the test, discard the reading.</p> <p>On each measure(within the product area):</p> <ul style="list-style-type: none"> • Front left, front right. • Back left, back right. <p>Record the temperatures °C Shelf temperatures may drift during the test so it is a good idea to take a reading of the shelf and the probe at the same time.</p> <table border="1" data-bbox="261 741 1318 1512"> <thead> <tr> <th>Probe Position</th> <th>Shelf No.</th> <th>Current Probe Reading</th> <th>Current Shelf Reading</th> <th>Probe Position</th> <th>Shelf No.</th> <th>Current Probe Reading</th> <th>Current Shelf Reading</th> </tr> </thead> <tbody> <tr> <td rowspan="5">Front Left</td> <td>1</td> <td></td> <td></td> <td rowspan="5">Front Right</td> <td>1</td> <td></td> <td></td> </tr> <tr> <td>2</td> <td></td> <td></td> <td>2</td> <td></td> <td></td> </tr> <tr> <td>3</td> <td></td> <td></td> <td>3</td> <td></td> <td></td> </tr> <tr> <td>4</td> <td></td> <td></td> <td>4</td> <td></td> <td></td> </tr> <tr> <td>5</td> <td></td> <td></td> <td>5</td> <td></td> <td></td> </tr> <tr> <td rowspan="5">Back Left</td> <td>1</td> <td></td> <td></td> <td rowspan="5">Back Right</td> <td>1</td> <td></td> <td></td> </tr> <tr> <td>2</td> <td></td> <td></td> <td>2</td> <td></td> <td></td> </tr> <tr> <td>3</td> <td></td> <td></td> <td>3</td> <td></td> <td></td> </tr> <tr> <td>4</td> <td></td> <td></td> <td>4</td> <td></td> <td></td> </tr> <tr> <td>5</td> <td></td> <td></td> <td>5</td> <td></td> <td></td> </tr> </tbody> </table>	Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading	Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading	Front Left	1			Front Right	1			2			2			3			3			4			4			5			5			Back Left	1			Back Right	1			2			2			3			3			4			4			5			5		
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<p>The table above will require setting up the mobile probes at least 5 times. Are the findings acceptable?</p> <p>If unacceptable temperature differences occur, contact Frozen in Time 01347 878158</p>	Y	N																																																																							

1i

Test the temperature at different levels throughout the operating range. As long as the operator/test person knows where the probes are positioned and the test time data can be taken directly from the data logging reporting data base. The reporting software can be transferred to a graph to show the dynamic, historical movement of the temperature traces. See section 6. See below extra tables for different levels

Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading	Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading
Front Left	1			Front Right	1		
	2				2		
	3				3		
	4				4		
	5				5		
Back Left	1			Back Right	1		
	2				2		
	3				3		
	4				4		
	5				5		

Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading	Probe Position	Shelf No.	Current Probe Reading	Current Shelf Reading
Front Left	1			Front Right	1		
	2				2		
	3				3		
	4				4		
	5				5		
Back Left	1			Back Right	1		
	2				2		
	3				3		
	4				4		
	5				5		

1m	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 0.05), 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 0.03 in 18 hours?	Y	N
1n	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 Trace 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	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 0.1mbar 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: 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	Y N/A	N

		<p>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 Frozen in Time</p>		
1i	Defrost	<p>With the refrigeration switched OFF, press the “defrost” button ON.</p> <p>Does the condenser chamber become warm does it reach its temperature set point.</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	<p>Have personnel that will use the HS series freeze drier been adequately trained?</p> <p>Are personnel familiar with: All the touch screen buttons and displays Condenser 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?</p>	Y	N
3b	User's Manual	Are the personnel who are to use or maintain the HS series able to locate the User's Manual for the machine?	Y	N

9.3. Summary

HS-2/HSL-2 Freezedrier 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