Operating Manual
Freeze Dryer

ALPHA 1-4 LSC

Part. No. 102041

ALPHA 2-4 LSC

Part. No. 102042

CHRIST

Freeze Dryers

Rotational-Vacuum-Concentrators

Version 09.2006 - English

OPERATING MANUAL

ALPHA 1-4 LSC / ALPHA 2-4 LSC

Order Number:

Serial Number:

In case of inquiries please state the above numbers.

For service please contact:

MARTIN CHRIST Gefriertrocknungsanlagen GmbH
An der unteren Söse 50, D-37520 Osterode
Phone +49 (0) 5522 / 5007-25, Fax +49 (0) 5522 / 5007-12
eMail: service@martinchrist.de

Preface

Dear customer

Congratulations on purchasing a CHRIST freeze dryer**.**

The freeze dryer **ALPHA 1-4 LSC / ALPHA 2-4 LSC** is equipped with a lot of user-friendly options which make the operation easier for you.

The newly designed **ALPHA 1-4 LSC / ALPHA 2-4 LSC** is a universally usable CHRIST freeze dryer for laboratories, R&D departments and scientific institutes. Our well proven range of accessories allows application-oriented configuration of the equipment for almost all types of drying processes in round bottom flasks, dishes, ampoules, injection bottles, etc.

A special advantage is the easy-to-use and self-explaining LSC control system (Lyo Screen Control):

- ¼ VGA-LC-Display
- graphical user interface with one knob operation („turn and push“)
- clearly arranged overview of process data in a freeze dryer flow sheet
- input of a maximum of 32 freeze drying programs in tabular form (optional)
- WINDOWS-NT-based software available.

Our new freeze dryer program offers a wide range of functions for a variety of practical applications.

We thank you for your confidence and wish you a successful application of your freeze dryer.

MARTIN CHRIST Gefriertrocknungsanlagen GmbH

An der unteren Söse 50, D-37520 Osterode

Phone +49 (0) 5522 / 5007-0, Fax +49 (0) 5522 / 5007-12,

Internet: www.martinchrist.de, eMail: info@martinchrist.de

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1.1. Introduction

What is freeze drying (lyophilisation)?

Freeze drying means: Extraction of water from frozen material. The drying process takes place by avoiding the liquid state through sublimation, i.e. direct conversion from ice to vapor. This happens under vacuum and the temperature in the product is normally 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 addition of water.

As the drying process takes place in the frozen state at very low temperatures it is possible, for example, to dry proteins which will not denature. Also most of the other chemical compounds will be qualitatively and quantitatively unchanged.

Through freeze drying the product, mainly of biological origin - such as tissues, tissue extracts, bacteria, vaccines and sera - is transformed into a dry product. During this process enzymatic, bacterial and chemical changes are largely avoided.

Freeze drying (lyophilisation) is the gentlest process for preserving the biological properties of sensitive tissue and tissue components.

Lyophilisation is also the best method when drying inorganic products – e.g. nanoscale dispersions – the particle surfaces of which should remain unchanged.

1.2. Areas of application

The freeze dryer **ALPHA 1-4 LSC / ALPHA 2-4 LSC** is a high-performance universal laboratory and pre-production unit for freeze drying of solid or liquid products in ampoules, vials, glass flasks, plasma bottles or dishes. All operations necessary for freeze drying can be realized in one and the same unit:

- Freezing of the products
- Freeze drying (sublimation) of the products at user-defined temperature limit values and pressure limit values
- Final drying of the products at user-defined temperature limits and high final vacuum for the removal of capillarily or molecularly bound water.

The freeze dryer **ALPHA 1-4 LSC / ALPHA 2-4 LSC** is suitable for drying bacteria and virus cultures, blood plasma, serum fractions, antibodies, sera, vaccines and pharmaceutical products such as chloramphenicol, streptomycin, vitamins, ferments as well as plant extracts for biochemical tests.

1.3. Technical data of freeze dryer ALPHA 1-4 LSC / ALPHA 2-4 LSC

Performance data	ALPHA 1-4 LSC	ALPHA 2-4 LSC
Ice condenser capacity:	4 kg max.	4 kg max.
Ice condenser performance[1)]:	4 kg/24 h max.	4 kg/24 h max.
Ice condenser temperature[1)]:	approx. –55°C	approx. –85°C
Possible shelf temperatures when freezing and drying inside the ice condenser chamber (process A) [1)]:	approx. -25 °C to +99°C	approx. -50°C to +99°C
Possible shelf temperatures when drying outside the ice condenser chamber (process B) [1)]:	room temperature to +99°C	room temperature to +99°C
Max. shelf surface area when drying inside the ice condenser chamber (process A):	1 shelf Ø200mm ≙ 0.031 m^2	1 shelf Ø200mm ≙ 0.031 m^2
Max. shelf surface area when drying outside the ice condenser chamber (process B):	10 shelves, Ø200mm ≙ 0.031 m^2 or 5 shelves, Ø375mm ≙ 0.110 m^2	10 shelves Ø200mm ≙ 0.031 m^2 or 5 shelves, Ø375mm ≙ 0.110 m^2
Max. shelf surface area when drying in glass vials with sealing under vacuum or nitrogen atmosphere outside the ice condenser chamber (process B):	4 shelves, Ø250mm ≙ 0.045 m^2	4 shelves, Ø250mm ≙ 0.045 m^2

Performance data	**ALPHA 1-4 LSC**	**ALPHA 2-4 LSC**
Drying in round-bottom flasks; please notice that the max. ice condenser capacity is 4 kg (process B):	12 pieces or 24 pieces	12 pieces or 24 pieces
Physical data (without vacuum pump):		
Dimensions of the unit:	width: 390 mm height: 415 mm depth: 555 mm (incl. vacuum flange connection)	width: 390 mm height: 415 mm depth: 555 mm (incl. vacuum flange connection)
Weight:	approx. 48 kg	approx. 60 kg
Noise emissions according to DIN 45635:	54 dB(A)	54 dB(A)
Electromagnetic compatibility according to EN 55011:	class B	class B
Filling quantities:		
Refrigerant:	see label on the back of the unit	see label on the back of the unit
Connection requirements with vacuum pump 0.4 KVA:		
Supply voltage:	230 V / 50 Hz (others upon request)	230 V / 50 Hz (others upon request)
Power rating:	1.6 KW	1.8 KW
Max. current consumption:	8.0 A	11.0 A

Performance data	ALPHA 1-4 LSC	ALPHA 2-4 LSC
Main fuse rating:	10.0 A	12.0 A
Ambient temperature[1)]:	+10°C to +25°C (higher temperatures upon request)	+10°C to +25°C (higher temperatures upon request)
Equipment connections:		
Vacuum connection:	Small flange connection DN 25KF (ISO 28403, DIN 2861)	Small flange connection DN 25KF (ISO 28403, DIN 2861)
Drain valve:	Hose nozzle DN10 (outside diameter 12 mm)	Hose nozzle DN10 (outside diameter 12 mm)
Aeration valve:	Hose nozzle DN4 (outside diameter 8 mm) max. 0.2 bar overpressure	Hose nozzle DN4 (outside diameter 8 mm) max. 0.2 bar overpressure

1) All machine specifications (especially for temperatures, power and capacity) refer to the nominal ambient temperature of 20°C

The scope of delivery includes:

- 1 tube of high-vacuum grease
- 1 liter of vacuum pump oil (only in case a pump is included)
- 1 set of flange components and several small parts for service and maintenance purposes
- 0.5 m drain hose (silicone 9 x 12 mm)
- 1 operating manual and further detailed technical documentation
- Commissioning of the unit (inside Germany)

The scope of delivery does not include:

- Installation of the exhaust pipe of the vacuum pump (not necessary when using an exhaust filter).

1.4. Standards and regulations

Please refer to the enclosed EU Declaration of Conformity.

1.5. Safety instructions

1.5.1. CAUTION! Disconnect the mains plug!

As current-carrying parts are accessible inside the unit the mains plug must be disconnected before the side panels or the rear panel are opened or before the control panel is removed.

For maintenance work the unit must be switched off with the mains switch.

1.5.2. CAUTION! Solvents!

Acidic products or products with a high solvent concentration cannot be dried without special protective measures and devices such as, for example, cooling trap for protection of the vacuum pump (if necessary check with our factory). The unit can also be damaged by corrosion.

Special caution is necessary when using azides because a dangerous explosive develops in combination with copper or non-ferrous metals! It is absolutely essential to consult our factory!

1.5.3. ADVICE! Cleaning and Maintenance of the Unit!

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

1.5.4. WARNING! Freezing of limbs to surfaces!

During operation of the freeze dryer, dangerous situations in the ice condenser chamber may arise. When putting in the shelves, make sure that no limbs come into contact with the condenser in the ice condenser chamber as the limbs may freeze to the surface. The limb can only be detached from the surface by applying heat. Liquid should not be used.

1.5.5. NOTE! Transport instructions!

Please keep the packaging for possible subsequent dispatch.

The freeze dryer should be carried by two persons by holding it underneath on both sides.

WARNING! When transporting or setting up the unit, do not hold the unit at the plastic control panel. When putting the unit down on a surface, there is a danger of squeezing hands or fingers.

Correct

Wrong

1.6. Prohibited freeze drying processes

1. Operation of the freeze dryer when it is not properly installed.
2. Operation of the freeze dryer without panels.
3. Operation of the freeze dryer by non-authorized personnel.
4. Operation of the freeze dryer when the shelves are not properly installed.
5. Operation of the freeze dryer with highly corrosive substances. It is not allowed to dry these substances, at least special safety measures have to be observed. The corrosive substances must not cause damage to material and or degrade the mechanical strength of the ice condenser chamber, the drying chamber, the lid or the accessory components.
6. Operation of the freeze dryer with accessories not allowed by the manufacturer, except for commercial freeze drying vessels made of glass or plastic. The user is explicitly warned against using equipment of low quality. Breaking glass or bursting vessels can cause dangerous situations during freeze drying.
7. Operation of the freeze dryer in hazardous locations.
8. Do not push or move the freeze dryer during operation. Do not lean on the freeze dryer.
9. Do not place potentially dangerous material, e.g. glass vessels containing liquids, near the freeze dryer.
10. Products which could react to the supply of high energy during the freeze drying process must not be dried.
11. Do not freeze-dry explosive or highly flammable substances.
12. Infectious, toxic, pathogenic and radioactive substances may only be dried in suitable vessels.
13. Substances which could damage the material of the shelves and of the chamber must not be dried or only if special safety measures are taken.

2. General Information on Freeze Drying

2.1. General Information on Freeze Drying

Freeze drying is the gentlest process for drying products. It is based on the physical phenomenon of sublimation, i.e. the direct conversion from solid to gaseous state. The frozen product is dried under vacuum without thawing. The ice condenser can also be described as a vapor pump as the moisture which evaporates under vacuum during drying freezes onto the ice condenser. Consequently, the vacuum pump is only intended to remove the air from the drying chamber (=gas pump) but not the vapor. In order to start the sublimation process, energy must be supplied to the product. This takes place during drying in round-bottom flasks or wide-neck filter bottles etc. due to the much warmer environment (direct heat contact), on unheated shelves by means of heat radiation from the environment and directly by means of the shelves when they are heatable. Once the “free water” has been completely removed from the product, it is also possible to remove the last traces of water bound by adsorption by means of a very deep vacuum. This part of the drying process is referred to as final drying (desorption).

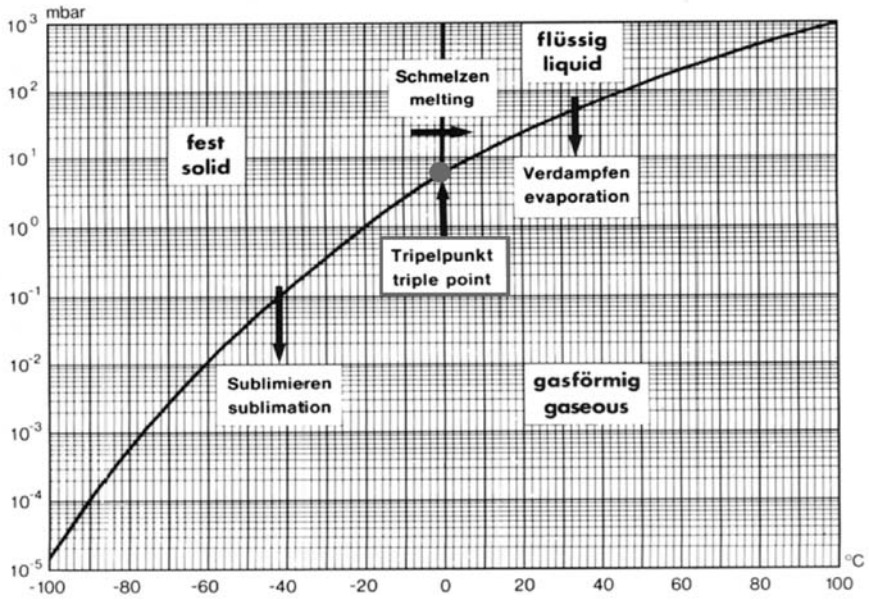
The main components of a freeze dryer are:

- Vacuum drying chamber with heating device
  a) heatable and unheatable shelves for drying in dishes
  b) shelves with sealing device for drying in bottles
  c) rubber valves for connecting round-bottom flasks, wide-neck filter bottles, etc.
  d) manifold for connecting round-bottom flasks, wide-, neck bottles, etc.
- Pumps to evacuate air and water vapor
  a) vacuum pump to evacuate the drying chamber **(= gas pump)**
  b) ice condenser with temperatures from -50°C to -105°C (depending on the type of unit) to remove the water vapor from the chamber **(= vapor pump)**

Sublimation

The principle of sublimation is briefly explained using the phase diagram of water (freeze drying of mainly aqueous solutions, see vapor pressure curve). If the atmospheric pressure is higher than 6.11 mbar, water passes through all three phases (solid, liquid, gas) when the temperature is lowered or raised. At 6.11 mbar the melting pressure curve, vapor pressure curve and sublimation pressure curve meet in one point called triple point. At this point, all three phases occur in parallel (simultaneously). Below this point, i.e. when the pressure is lower than 6.11 mbar, the ice is converted directly from a solid to a gaseous phase on reaching the sublimation pressure curve (vapor pressure curve above ice).

Vapor pressure curve for ice and water



1mbar = 10^2Pa

Conversion table "Vapor pressure above ice" (sublimation curve)

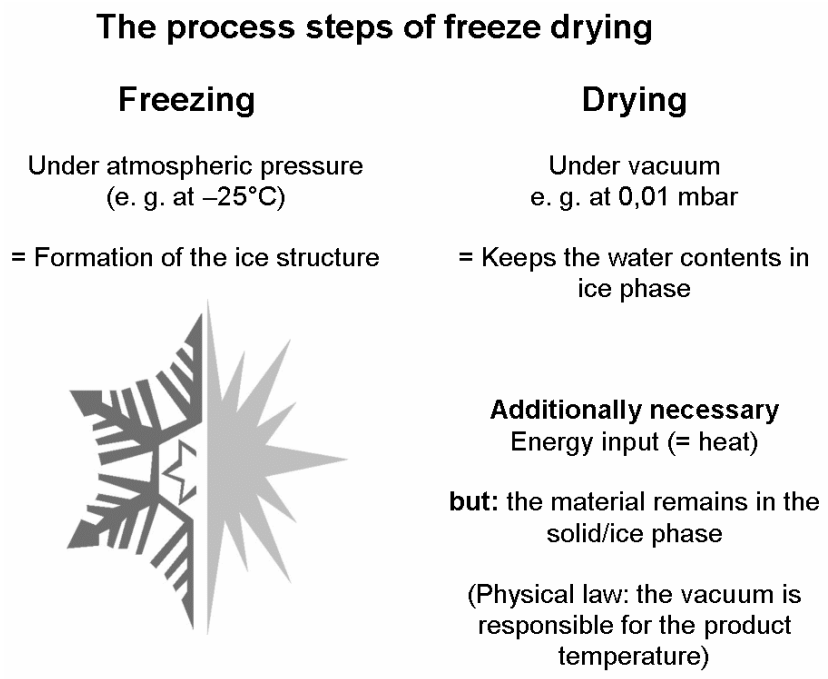
Pressure units: 1mbar = 100Pa
1Pa = 0.01mbar

Temperature units:

$$T = t + 273$$
$$t = T - 273$$
$$t_F = 1.8 \cdot t + 32$$
$$t = \frac{t_F - 32}{1.8}$$

T = thermodynamic temperature K (Kelvin)
t = temperature in degree Celsius °C
t_F = temperature in degree Fahrenheit °F

°C	≙ mbar	°C	≙ mbar	°C	≙ mbar	°C	≙ mbar
0	6.110	-20	1.030	-40	0.120	-60	0.011
-1	5.620	-21	0.940	-41	0.110	-61	0.009
-2	5.170	-22	0.850	-42	0.100	-62	0.008
-3	4.760	-23	0.770	-43	0.090	-63	0.007
-4	4.370	-24	0.700	-44	0.080	-64	0.006
-5	4.020	-25	0.630	-45	0.070	-65	0.0054
-6	3.690	-26	0.570	-46	0.060	-66	0.0047
-7	3.380	-27	0.520	-47	0.055	-67	0.0041
-8	3.010	-28	0.470	-48	0.050	-68	0.0035
-9	2.840	-29	0.420	-49	0.045	-69	0.0030
-10	2.560	-30	0.370	-50	0.040	-70	0.0026
-11	2.380	-31	0.340	-51	0.035	-71	0.0023
-11	2.170	-32	0.310	-52	0.030	-72	0.0019
-13	1.980	-33	0.280	-53	0.025	-73	0.0017
-14	1.810	-34	0.250	-54	0.024	-74	0.0014
-15	1.650	-35	0.220	-55	0.021	-75	0.0012
-16	1.510	-36	0.200	-56	0.018	-76	0.0010
-17	1.370	-37	0.180	-57	0.016	-77	
-18	1.250	-38	0.160	-58	0.014	-78	
-19	1.140	-39	0.140	-59	0.012	-79	



2.2. Preparation

In case the unit is equipped with a pressure control valve the vacuum pump should be warmed up. The operating temperature should be reached before loading the vacuum pump with condensable gases. In this way, the service life of the vacuum pump can be considerably extended.

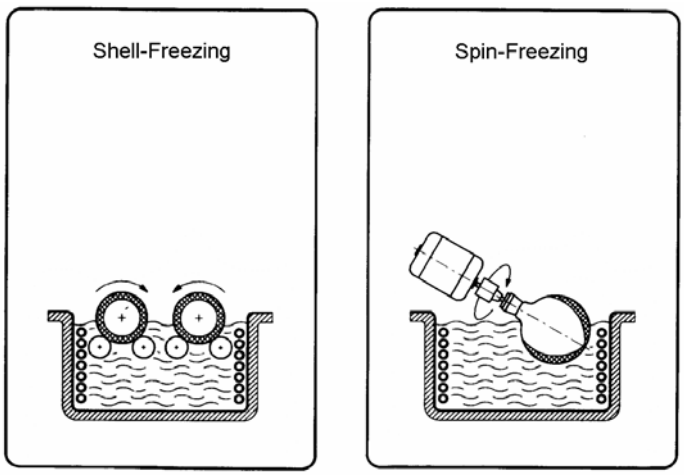
The vacuum pump can be operated already during the freezing process when the pressure control valve is closed. The vacuum pump should be warmed up for at least 15 minutes or it should be switched on at least 15 minutes before starting the main drying process.

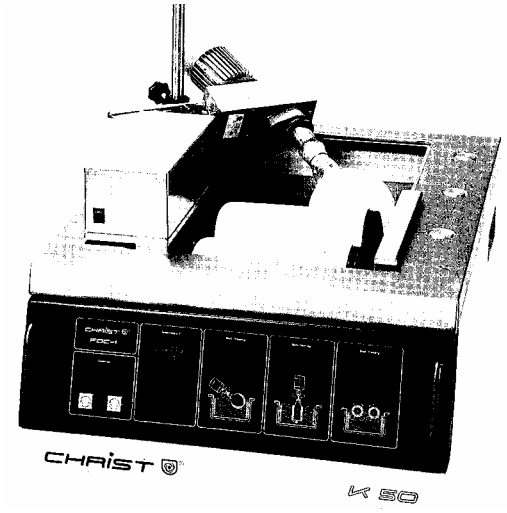
It may occur that the vacuum in the ice condenser chamber or in the drying chamber goes up during the main drying process (e.g. from 0.63 mbar to 0.47 mbar) although the valve to the vacuum pump is closed. This can be explained physically by the pumping effect of the ice condenser (“Cryo Pumping Effect”).

2.3. Freezing

Small product quantities are frozen directly inside the ice condenser chamber of the **ALPHA 1-4 LSC / ALPHA 2-4 LSC**. Larger quantities are pre-frozen in a deep-freeze.

If liquids are to be dried in bottles with a layer thickness of more than 1 cm we recommend that pre-freezing is carried out with a shell or spin freezing device in a cooling bath (see picture). Due to the centrifugal force the liquid to be frozen will rise on the wall of the bottle and freeze. With this freezing process the layer thickness is reduced and thus the total drying period is shortened considerably.





Freezing inside the unit is not necessary if the product is pre-frozen or stored in e. g. a deep-freeze. In this case, especially when freeze drying small quantities, it is advisable to pre-cool the shelves in order to avoid partial thawing during the evacuation.

Possible water residue must be removed from the ice condenser chamber. The drain valve and the aeration valve are closed.

The ground-in stopper of the drying chamber must be greased with high-vacuum grease!

The layer thickness of the product should not exceed 1 to 2 cm as otherwise this has a negative effect on the duration of the drying process.

2.4. Main drying

The vacuum pump is switched on.

Please note:

Defrosting during the drying process is possible (visible foaming) when drying products containing e. g. solvents or high salt concentrations. In this case it is necessary to freeze the product at temperatures as low as possible, e.g. in liquid nitrogen.

Warning:

Acidic products or products with a high solvent concentration cannot be dried without special safety measures and devices, such as an additional LN_2 cooling trap for protection of the vacuum pump (if necessary contact our company). Another possibility is to use chemical-resistant vacuum hybrid pumps (e.g. RC-5).

Special precautions are necessary when using azides because a dangerous explosive develops in combination with copper or non-ferrous metals! It is absolutely essential to consult our company!

As soon as sublimation of the water vapor from the frozen product begins, heat is extracted and consequently the product continues to cool down.

The maximum rate of sublimation is reached at the start of the drying process.

Depending on the rate of sublimation the ice condenser temperature and thus the pressure in the drying chamber or ice condenser chamber rises.

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 pressure inside the drying chamber during the drying process.

With increasing pressure (not vacuum!) the rate of sublimation rises and the drying period is shortened.

The water vapor generated during the main drying phase is not pumped off by the vacuum pump but 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 are also pumped off by the vacuum pump. Therefore, the vacuum pump is equipped with a gas ballast device.

If the gas ballast valve is open, the extracted condensable vapors will be emitted via the exhaust pipe together with air.

For this reason the gas ballast valve must be open during the main drying phase!

The gas ballast valve can only be closed for final drying since there is not much water vapor left during this section of the drying process.

During the main drying phase the moisture is removed by sublimation, during final drying the bound moisture is removed by desorption.

This small quantity of water vapor generated during the final drying phase can be pumped off by the vacuum pump even when the gas ballast valve is closed (for some hours).

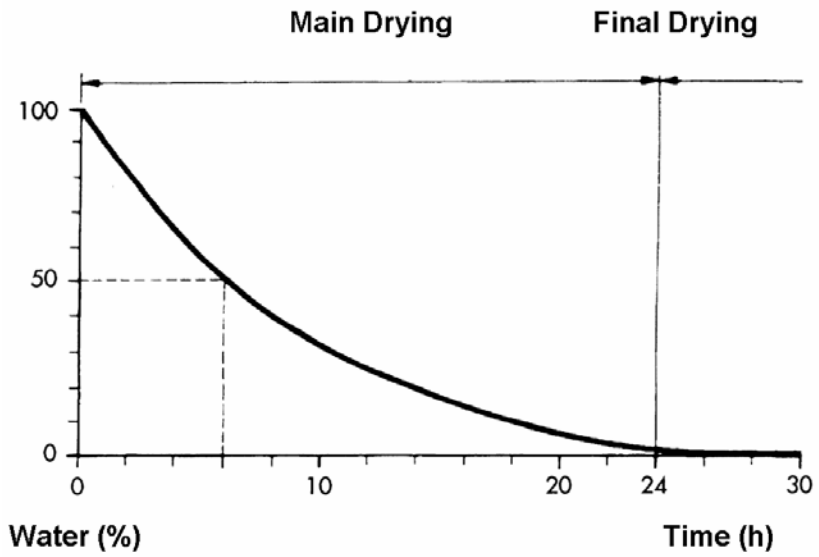
In general, operation with closed gas ballast valve is not necessary. The recommended vacuum pump reaches with open gas ballast valve a final pressure which corresponds to the water vapor partial pressure that can be reached.

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 main drying phase is reached, when the product temperature is nearly the same as the shelf temperature (temperature difference between shelf and product approx. 3 K to 5 K). If the adsorptively bound water is to be removed from the product, the final drying phase can be started.

The following picture shows the drying process for a product containing approx. 10 % solid matter. During the first quarter of the main drying phase 50 % of the water content is condensed. During the next quarter of the main 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 water vapor still to be extracted has to pass through the already dried layers. During the drying process the inner resistance increases. Thus the drying curve is primarily determined by the latent heat of sublimation and the water vapor transport speed. In order to increase the specific heat conduction properties of the product to be dried and to keep the water vapor volume as low as possible it is necessary that drying takes place as close as possible to the solidification point (eutectic point).



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

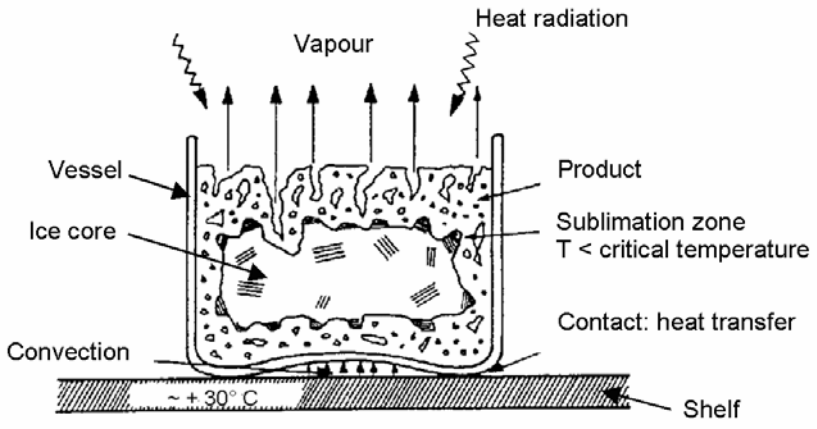
Interesting correlations:

1.0	gram of ice at		
1.0	mbar assumes a volume of	1	m^3 vapor
0.1	mbar assumes a volume of	10	m^3 vapor
0.01	mbar assumes a volume of	100	m^3 vapor

Heat supply during drying

The required heat supply to the product to be dried takes place through direct heat contact in the drying chamber, heat conduction through gas or through radiation. Heat transfer by direct contact and heat conduction through gas are the most usual sources of heat in today's freeze dryers. The constraints caused by the former can be seen in the following diagram.

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 vessel and/or by convection via the shelf and vessel or product.

At the beginning of sublimation the transfer of heat is very effective from the wall of the vessel to the frozen product. However, soon an area develops which is ice-free, porous and dried and has a corresponding temperature gradient between the wall of the vessel and the product. The poor heat conductivity of the already dried product can lead to an increase in temperature of the ice core. If the core temperature rises above the solidification temperature, the product begins to thaw. 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.

2.5. Final 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

0.011 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 0.120 mbar.

The final pressure measured by the vacuum sensor when there is no product in the unit and its corresponding ice temperature value is mainly determined by the warmest place of the ice on the ice condenser chamber. Moreover, this value is affected by residues or parts of solvents in the product with a higher vapor pressure.

2.6. End of drying and aeration

A rough indication of the end of drying is the vacuum and the ice condenser temperature. The ice condenser is no longer loaded and reaches the final temperature of approx. -55°C to -85°C. The pressure in the drying chamber decreases according to the ice condenser temperature.

The end of the drying process is reached when the temperature of the product and that of the shelves are about 15 to 20°C. At the same time, both temperatures should not deviate by more than 5 K from each other.

The vacuum pump is switched off and the drying chamber is aerated via a rubber valve or the aeration valve. The aeration valve can also be used to “flood” the unit with nitrogen or another inert gas instead of using air.

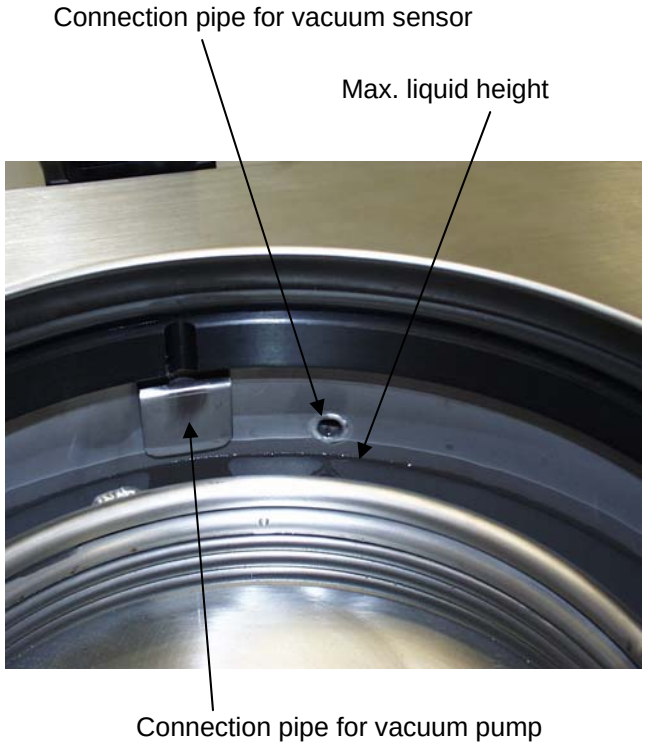
Then the unit is switched off and the product is removed.

2.7. Defrosting

Defrosting of the ice condenser is carried out at room temperature or with warm water. At a maximum the ice condenser chamber may be half filled with water.

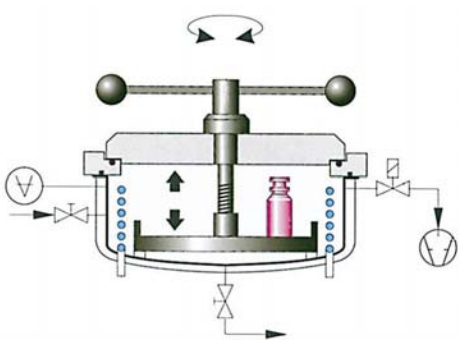
When defrosting the ice condenser with (warm) water, it is of crucial importance that no water gets into the pipe connection for the vacuum pump and the vacuum sensor (see figure)!

The condensate as well as the defrosting water is drained through the drain valve at the left side of the unit. To do this, a hose is put onto the nozzle. The condensate and the defrosting water are collected in a vessel.



3. Description of the Freeze Drying Processes

3.1. Freezing and drying inside the ice condenser chamber (process A) on a temperature-controlled shelf



First, possible water residue is removed from the ice condenser chamber. The drain valve is opened so that the water residue can drain off. The ice condenser chamber should be wiped out if necessary.

The refrigeration unit and the vacuum pump are activated via the control system to pre-cool the ice condenser and to warm up the vacuum pump. The valve is closed during the warm-up of the vacuum pump. In case the unit is not equipped with an electromagnetic pressure control valve, we recommend using a manual stop valve. Without such a valve, the vacuum pump cannot be warmed up.

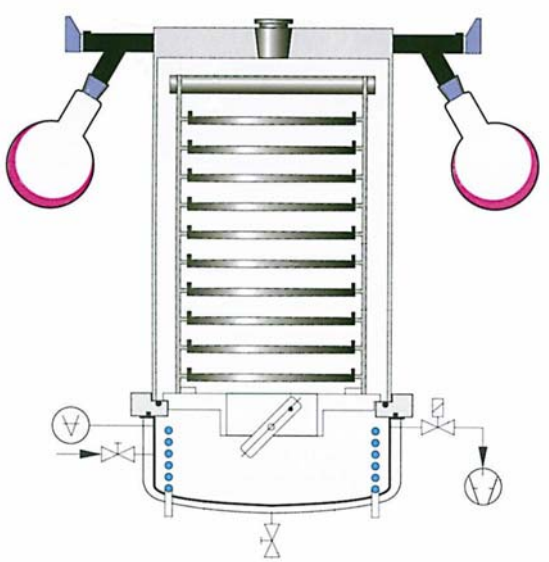
With the sealing device (option), injection bottles can be sealed with serrated rubber stoppers inside the ice condenser chamber under vacuum or inert gas. To do this, the shelves are moved against each other by a pressure plate using a spindle. (When drying inside the ice condenser chamber, only 1 shelf can be used. When drying outside the ice condenser chamber, a maximum of 4 shelves can be used.)

After completion of the drying process, the lever of the rotary leadthrough is turned to the right until a resistance is felt.

In order to seal the bottles, the shelf must be completely filled. For small quantities, at least three spacers must be distributed evenly on each shelf (according to the height of the bottles with inserted rubber stoppers).

Suitable spacers are available upon request.

3.2. Separate freezing and drying outside the ice condenser chamber (process B) on temperature-controlled shelves



The product can also be dried outside the ice condenser chamber. In this case, the product has to be frozen first, e.g. in a deep-freeze.

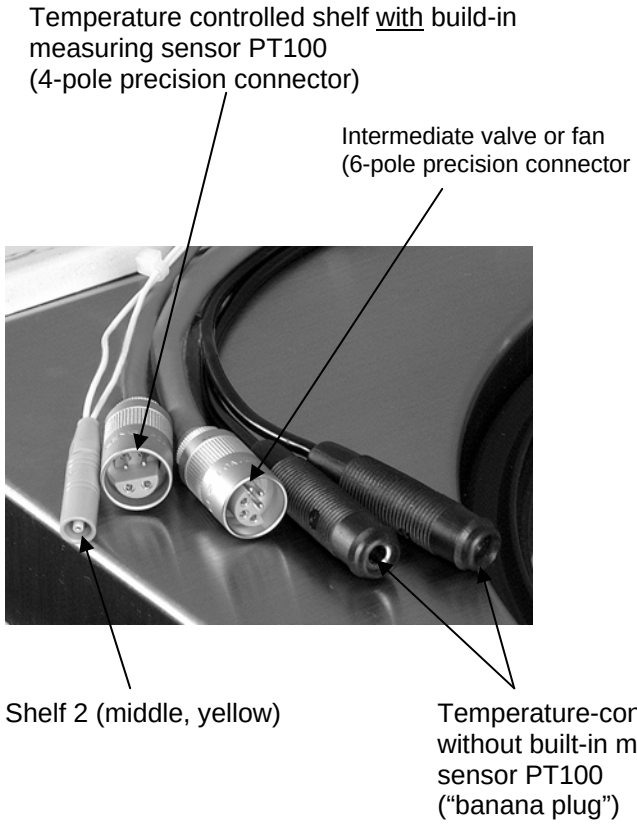
Possible water residue is removed from the ice condenser chamber. The drain valve is opened so that the water residue can drain off. The ice condenser chamber should be wiped out if necessary.

The refrigeration unit and the vacuum pump are activated via the control system to pre-cool the ice condenser and to warm up the vacuum pump. The valve is closed during the warm-up of the vacuum pump. In case the unit is not equipped with an electromagnetic pressure control valve, we recommend using a manual stop valve. Without such a valve, the vacuum pump cannot be warmed up.

The rack is set onto the base plate or onto the intermediate valve with a base plate. In the case of small samples, it is recommended to pre-cool the shelves in order to avoid partial thawing during the evacuation.

The heating current is supplied to the shelves via two connection cables with banana plugs. The connection cables are plugged into the two sockets of the electrical leadthrough and into the holes in the rack or in the sealing device.

To control the shelf temperature, **one shelf** with a measuring sensor has to be connected. The plug of the shelf with the measuring sensor has to be connected to the socket of the vacuum-tight electrical leadthrough.



All shelves are supplied with the same heating power. It is not possible to consider different heat requirements of the upper and lower shelves. Thus, slight differences in temperature between the shelves cannot be avoided.

When freezing and drying is performed separately outside the ice condenser chamber, the system starts directly with the main drying phase. In case the unit is equipped with a pressure control valve, the vacuum pump is activated and warmed up.

As soon as the shelves with the pre-frozen samples have been put into the unit, the unit and the drying chamber are sealed.

The main drying process has to be activated in the control system so that the pressure control valve opens and the main drying phase starts.

When the pressure falls below the pre-selected „**Heater safety pressure“**, the heater of the shelves is activated. The heater remains on until the nominal value of the shelf is reached.

3.3. Separate freezing and drying outside the ice condenser chamber (process B) with sealing device

Possible water residue is removed from the ice condenser chamber. The drain valve is opened so that the water residue can drain off. The ice condenser chamber should be wiped out if necessary.

The refrigeration unit and the vacuum pump are activated via the control system to pre-cool the ice condenser and to warm up the vacuum pump. The valve is closed during the warm-up of the vacuum pump. In case the unit is not equipped with an electromagnetic pressure control valve, we recommend using a manual stop valve. Without such a valve, the vacuum pump cannot be warmed up.

The rack is set onto the base plate or onto the intermediate valve with a base plate.

The sealing device is used to seal injection bottles under vacuum or inert gas with serrated rubber stoppers. Depending on the type of sealing device, the bottles are sealed on 1 to 2 shelves or on 1 to 4 shelves. To do this, the shelves are moved against each other by a pressure plate using a spindle.

The height of the pressure plate must be adjusted according to the height of the bottles. To do so, the threaded pin for the height adjustment is removed. The threaded rod is screwed into the lower shelf until its slotted head is on the same level as the rack (upper edge).

Then the pressure plate is fastened with the threaded pin in such a way that it will rest on top of the rubber stopper or slightly above it. When using two or more shelves, every additional shelf is set directly onto the rubber stoppers or slightly above them, just like the pressure plate.

Instead of the stopper, the vacuum-tight rotary leadthrough is put into the internal ground-in connection of the drying chamber. Before the insertion, the ground-in surface of the leadthrough is greased with vacuum grease.

After completion of the drying process the lever of the rotary leadthrough is turned to the right until a resistance is felt.

In order to seal the bottles, the shelf must be completely filled. For small quantities, at least three spacers must be distributed evenly on each shelf (according to the height of the bottles with inserted rubber stoppers).

Suitable spacers are available upon request.

3.4. Separate freezing and drying of liquids in flasks (process B)

Possible water residue is removed from the ice condenser chamber. The drain valve is opened so that the water residue can drain off. The ice condenser chamber should be wiped out if necessary.

The refrigeration unit and the vacuum pump are activated via the control system to pre-cool the ice condenser and to warm up the vacuum pump. The valve is closed during the warm-up of the vacuum pump. In case the unit is not equipped with an electromagnetic pressure control valve, we recommend using a manual stop valve. Without such a valve, the vacuum pump cannot be warmed up.

Several manifolds and attachable drying chambers with connections for rubber valves are available for drying outside the ice condenser chamber (see accessories catalogue). The drying chamber with connections for rubber valves is mounted directly on the sealing ring in the base plate. Manifolds with standard ground joints NS 45/40 are connected via the internal ground-in connection of the acrylic lid.

In order to ensure a vacuum-tight connection and to simplify the removal of the accessories, the ground-in surfaces must be slightly greased with vacuum grease before use. The accessory is then attached and turned by 360° for an even distribution of the grease.

The ground-in surfaces have to be cleaned and greased before any further installation of accessories.

Before the drying process can start, it is necessary to check that all valves are closed.

The main drying process has to be activated in the control system so that the pressure control valve opens and the main drying phase starts.

Attention:

The frozen samples can only be connected to the valves when the pressure has fallen below 1.030 mbar.

Liquids are frozen in flasks according to the shell freezing principle, by hand or with a rotator. These freezing methods reduce the layer thickness and thus also the drying time to a considerable extent.

Most of the available manifolds and valves allow a continuous connection and removal of flasks during the drying process. Every connection has a stop valve and an aeration valve.

If the rubber valves or stainless steel valves are stiff, they must be dismantled, cleaned, slightly greased with vacuum grease and reassembled.

It is also possible to connect a distributor for 15 ampoules to every rubber valve.

Using a distributor, a maximum of 15 ampoules can be simultaneously frozen in the cooling bath and connected to the manifold.

3.5. Separate freezing and drying of liquids in ampoules (process B)

The refrigeration unit and the vacuum pump are activated via the control system to pre-cool the ice condenser and to warm up the vacuum pump. The valve is closed during the warm-up of the vacuum pump. In case the unit is not equipped with an electromagnetic pressure control valve, we recommend using a manual stop valve. Without such a valve, the vacuum pump cannot be warmed up.

The manifold is equipped with blind plugs for the connection of a maximum of 48 ampoules. The blind plugs ensure that the manifold can be pre-evacuated.

The first hose is clamped in the middle with the supplied hose clamp and the blind plug is removed. The vacuum of the system is preserved.

The liquid in the ampoule is either frozen under rotation in a cooling bath or in a deep-freeze.

If shock-freezing is required, we recommend that freezing is carried out in liquid nitrogen or in a cooling bath.

The ampoule is then connected to the hose and the hose clamp is removed. Thus the partial pressure in the ampoule is suddenly decreased and partial thawing during the evacuation process is avoided.

One ampoule after the other is connected using this method.

The ampoules are sealed in the same way. The hose of the ampoule to be sealed is clamped and the ampoule is sealed under vacuum using the blow lamp.

If the ampoule breaks during sealing, the vacuum in the drying chamber is not affected since the hose is clamped with the hose clamp.

The remaining glass of the sealed ampoule is removed and the hose is closed with a blind plug.

In this way, one ampoule after the other can be sealed or new ampoules can be connected.

4. Installation and Commissioning of the Unit

4.1. Site of installation

ATTENTION! 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. +10°C and +25°C.

The refrigeration compressor of the freeze dryer is air-cooled. Sufficient air circulation must be ensured. A distance of at least 30 cm to the wall should be kept. The unit should not be positioned near radiators or heat sources and direct insolation must be avoided.

In 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 a failure of the refrigeration unit.

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.

CHRIST freeze dryers are units of safety class I. The units **ALPHA 1-4 LSC / ALPHA 2-4 LSC** are equipped with a three-wire connection cable with a 230VAC safety plug with ground contact.

4.3. Fuses on site

The freeze dryer must be protected with a 16 AG fuse.

4.4. Checking the ground connection

To check the ground conductor, there is an equipotential bonding screw at the rear panel of the freeze dryer. A ground conductor check can be carried out using a suitable measuring instrument.

4.5. Aeration

The aeration valve at the upper left side of the unit is used to aerate the ice condenser chamber.

If penicillin bottles have to be sealed under nitrogen and not in a vacuum, the ice condenser chamber can be flooded with nitrogen via the hose nozzle of the aeration valve.

CAUTION! Max. 0.2bar overpressure!

4.6. Condensate and defrosting water

The condensate and the defrosting water are discharged through the drain valve at the lower left side of the unit. Connect the supplied hose to the hose nozzle. The condensate and the defrosting water are collected in a vessel.

The condensate and the defrosting water can also be directly fed into a drain using this hose provided it can flow off freely. To achieve this, the hose must have a steady downward slope. It must be ensured that water does not collect in any part of the hose. The end of the hose always has to be above the level of the liquid in the collecting vessel. Otherwise there is the risk of water and dirt residues being sucked into the condenser chamber if there is a negative pressure when the drain valve is opened.

4.7. Vacuum pump exhaust gases

During the main drying phase, the separate vacuum pump must be operated with open gas ballast valve. The oil mist that is produced is normally trapped in an exhaust filter. If this is not the case, the oil mist has to be discharged.

A ½" hose can be connected to the exhaust flange of the vacuum pump RZ-2 or RC-5 and a ¾" hose can be connected to the exhaust flange of the vacuum pump DUO 5 or DUO 10. The hose either leads into the open air or into a vent.

During installation of the pipe special care must be taken that condensate cannot flow back into the pump. With upward leading pipes it is safest to use a separator (Woulfe's bottle or wash bottle) in the pipe.

We strictly recommend using an exhaust filter (oil mist separator). This filter prevents air pollution by oil mist which is emitted by the vacuum pump in varying quantities depending on the working pressure.

The filter is fastened to the exhaust flange of the vacuum pump.

The filter is equipped with a pressure relief valve indicating the saturation of the filter. The filter cartridge has to be cleaned or replaced at the latest when the pressure relief valve is activated. The collected oil is visible in the sight glass and discharged via the discharging screw.

Since spring 2004, most of the vacuum pumps delivered by CHRIST have been equipped with an automatic oil recirculation system. This system reuses the oil separated by the exhaust filter to feed the pump. A manual removal is no longer necessary.

(Please refer to separate operating manual of the vacuum pump!)

4.8. Initial start-up

ATTENTION! Prior to start-up, make sure that the freeze dryer has been properly set up and installed (see section 4.1 “Site of installation” and the following points).

4.8.1. Functional components and control elements

Aeration valve

Drying chamber Ø300

Ice condenser chamber

Ice condenser

Control system LSC

Mains switch

Additional interface connection (option, sep. accessory)

Serial interface

Vacuum pump power supply connection

Power supply connection of pressure control valve and stop valve

Mains fuse

Mains cable

Equipotential bonding screw for protective ground wire check and central ground connection

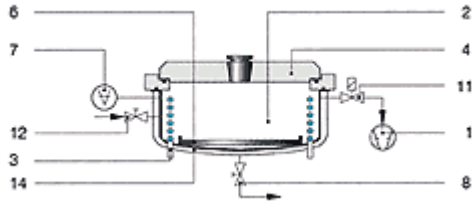
Name plate

Vacuum connection

Heat exchanger of refrigeration unit

4.8.2. Vacuum sensor, vacuum pump and pressure control valve connection

Connection diagram

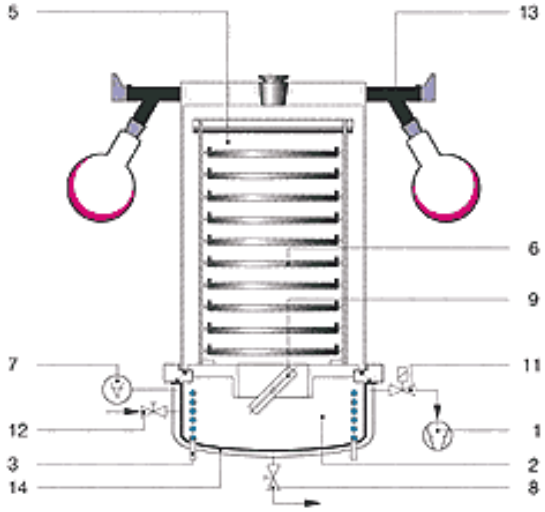


Process A (single-chamber system):

Freezing and gentle drying of low-freezing and thermolabile substances on cooled shelves inside the ice condenser chamber (see figure 1).

Typical drying vessels::

- Dishes
- Injection vials (can be sealed under vacuum)



Process B (double-chamber system):

Separate freezing (e.g. in a deep-freeze) and drying outside the ice condenser chamber (see figure 2)

Typical drying vessels:

- Dishes
- Injection vials (can be sealed under vacuum)
- Round-bottom flasks, wide-neck filter bottles
- Ampoules

1 Vacuum pump
2 Ice condenser chamber
3 Ice condenser
4 Glass lid
5 Drying chamber
6 Heatable shelf
7 Vacuum sensor
8 Drain valve
9 Motor- driven intermediate valve
11 Pressure control valve
12 Aeration valve
13 Rubber valve
14 Thermal insulation

The accessory components are connected to the ALPHA 1-4 LSC / ALPHA 2-4 LSC freeze dryer according to the connection diagram. Please observe the following:

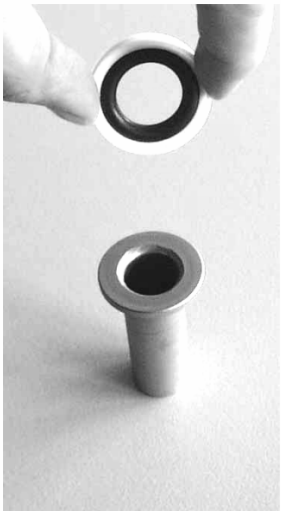
Centering rings and clamping rings with wing nuts are used as connecting elements (small flange connections in accordance with ISO 28403 and DIN 2861, see also the following instructions).

Information concerning the connection with centering rings and clamping rings

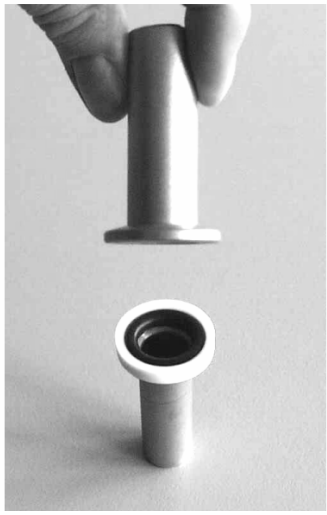
Small flange connections between aggregates or hose connections that are not mounted properly are often the reason for serious vacuum problems.

Please loosen the connection and replace the centering ring (with sealing ring inside) centrically between the flange connections. Seal the connection with the clamping ring by fastening the wing nut.

Please take care that the centering ring does not slip out of place and that is does not get tilted.

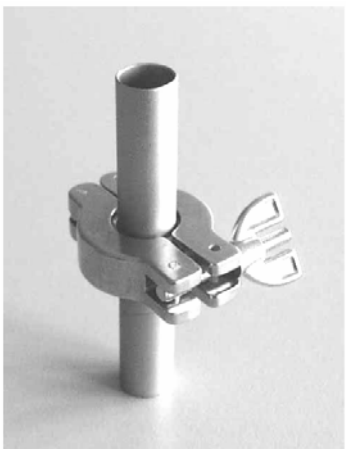


Small flange and centering ring



Small flange with centering ring and small flange

Mounting of the clamping ring



Fastening of the clamping ring

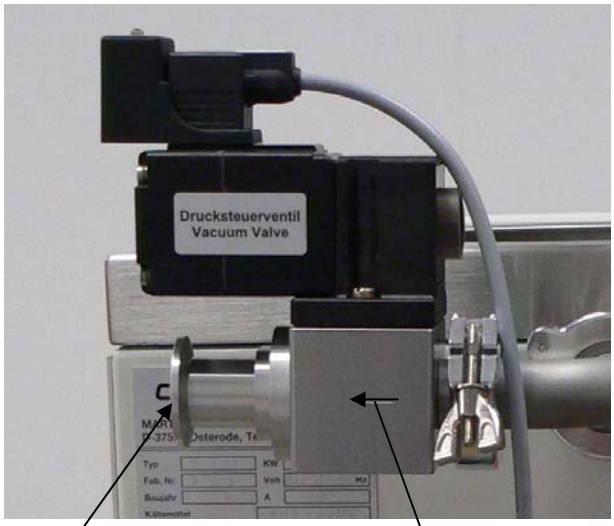
Installation of the VSP62 vacuum sensor

The vacuum sensor has to be connected to the vacuum connection at the back of the unit in an upright position using a centering ring and a clamping ring. The sensor connecting cable has to be connected to the corresponding socket at the back of the unit.

After power-on, the vacuum sensor needs a few minutes to reach its operating temperature.

Installation of the pressure control valve

If a pressure control valve is available, it has to be mounted between the freeze dryer and the vacuum pump. Then it has to be plugged into the power socket at the back panel of the unit.



Installation of the vacuum pump

The vacuum pump has to be connected to the unit and to the socket at the back panel of the unit.

Make sure that the vacuum exhaust gases are filtered or carried off.

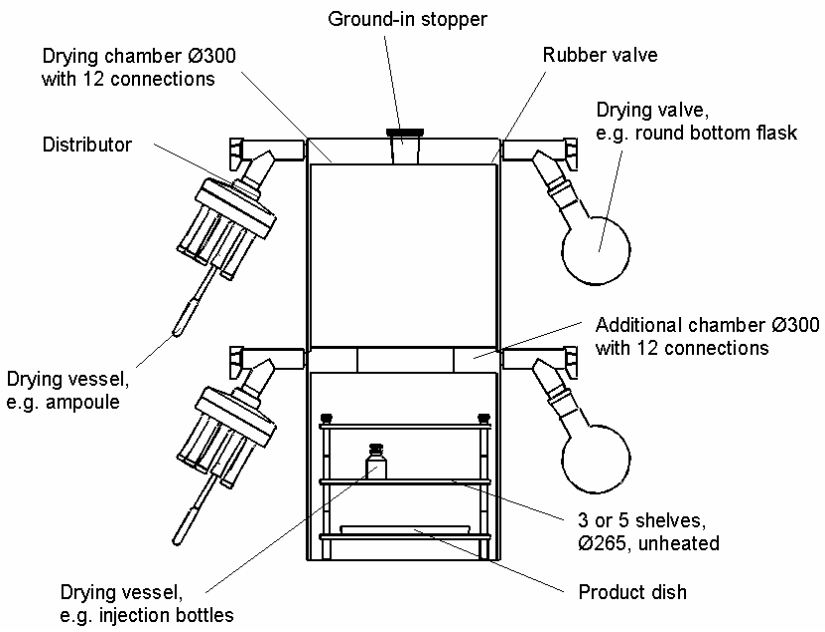
4.8.3. Installation of accessory components

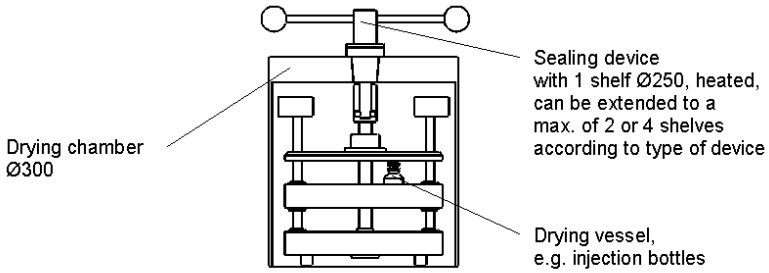
Further accessories (e.g. shelves, connections for round-bottom flasks) are added in accordance with the scope of supply.

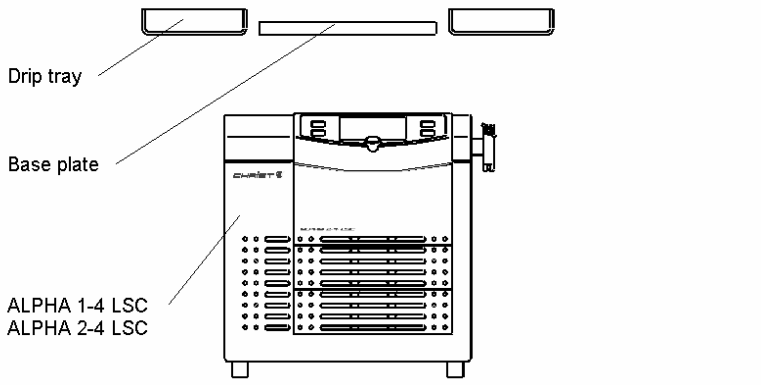
The drying chamber or a lid is placed on top of the ice condenser chamber.

Accessories

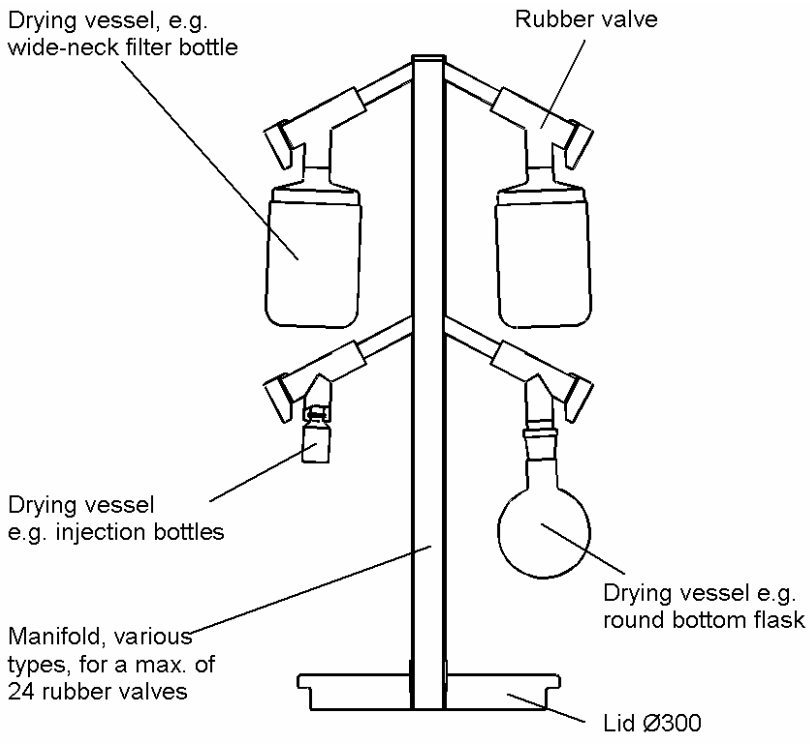
Process B

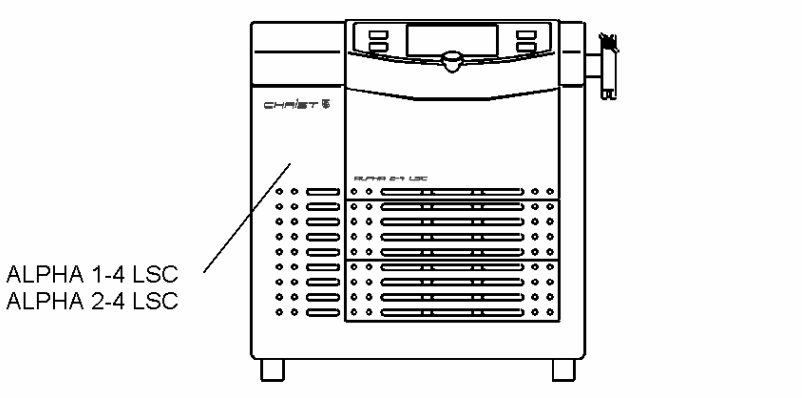




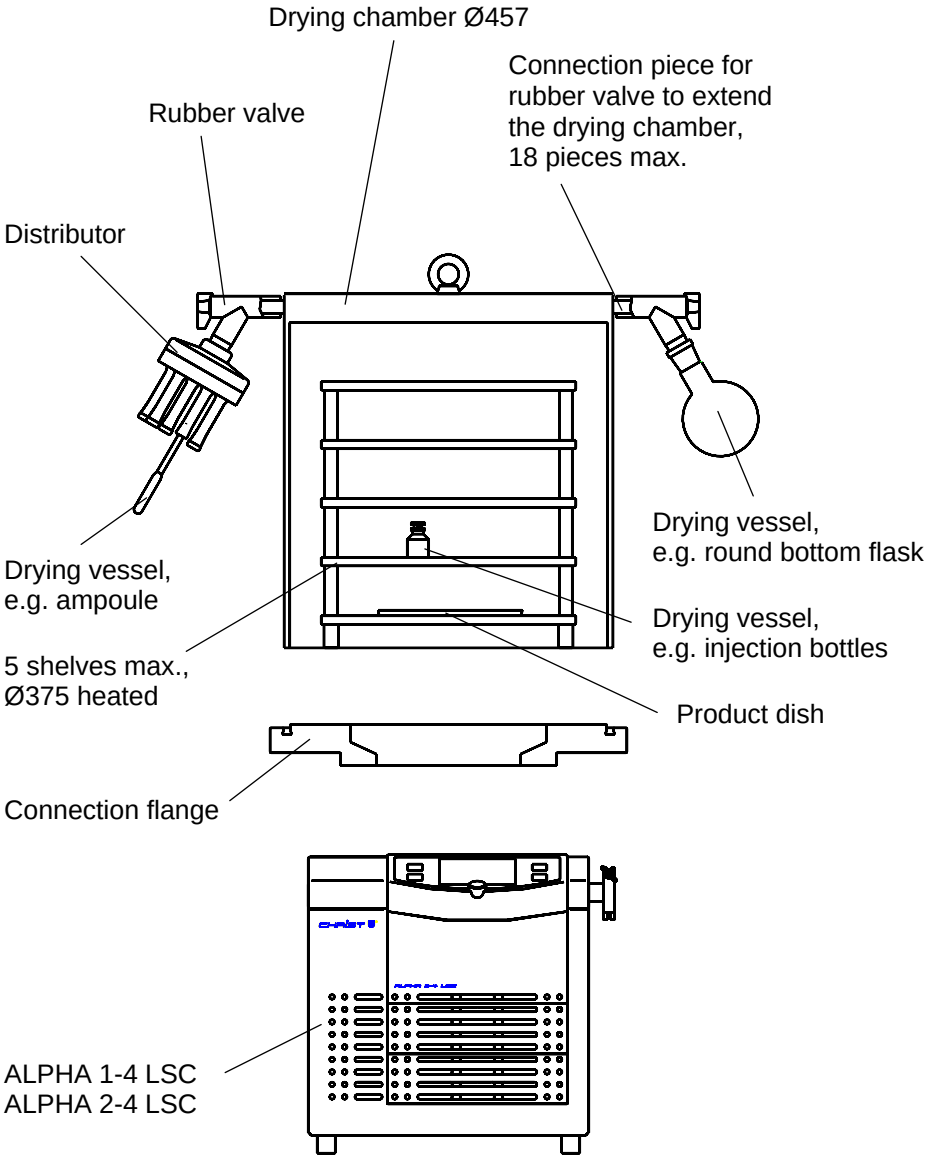


Process B

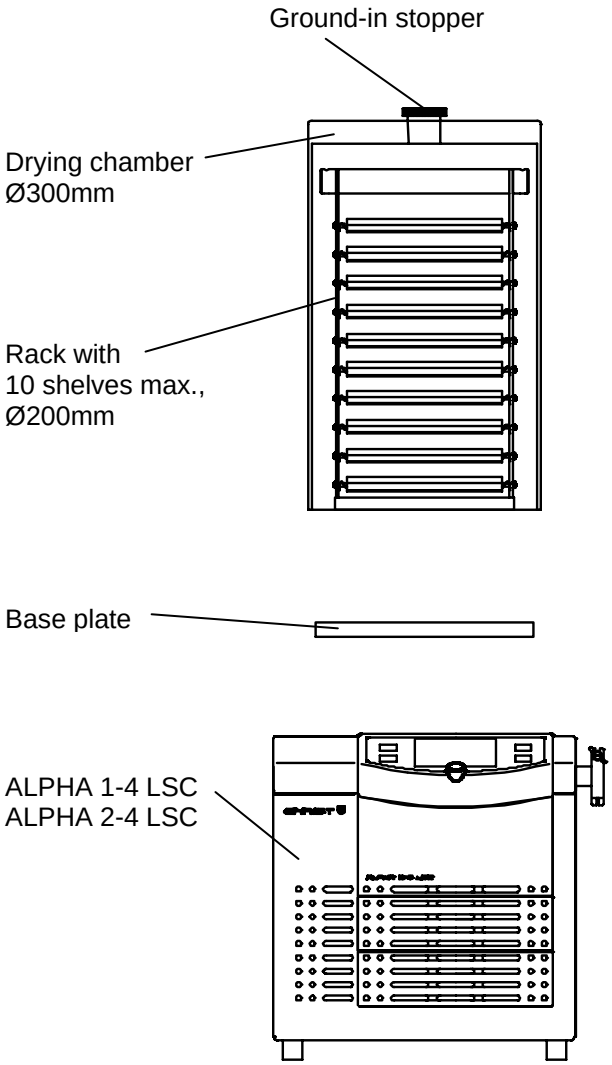

Drying vessel, e.g.
wide-neck filter bottle
Rubber valve
Drying vessel
e.g. injection bottles
Drying vessel e.g.
round bottom flask
Manifold, various
types, for a max. of
24 rubber valves
Lid Ø300


ALPHA 1-4 LSC
ALPHA 2-4 LSC

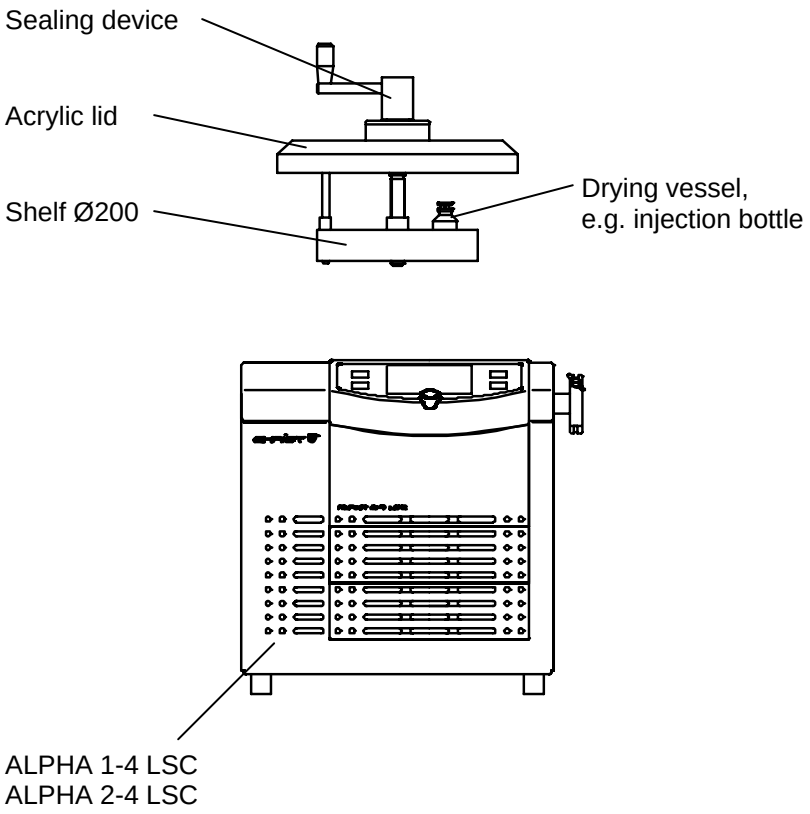
Process B


Drying chamber Ø457
Connection piece for rubber valve to extend the drying chamber, 18 pieces max.
Rubber valve
Distributor
Drying vessel, e.g. round bottom flask
Drying vessel, e.g. ampoule
Drying vessel, e.g. injection bottles
5 shelves max., Ø375 heated
Product dish
Connection flange
ALPHA 1-4 LSC
ALPHA 2-4 LSC

Process B


Ground-in stopper
Drying chamber
Ø300mm
Rack with
10 shelves max.,
Ø200mm
Base plate
ALPHA 1-4 LSC
ALPHA 2-4 LSC

Process A



4.8.4. Preparations for freeze-drying

Close the aeration valve and the drain valve on the left side of the unit.

If rubber valves are included in the system (located at the drying chamber), they have to be closed.

4.8.5. Power-on

Press the mains switch on the right side of the unit to switch the unit on.

At first, the LSC control system performs a self-test. This may take several seconds.

5. LSC Control System

5.1. Introduction

LSC (Lyo Screen Control) stands for a graphical user interface that is used to control freeze drying processes. The operation of the unit is very easy and self-explaining with the “turn & push” button. Most of the process data are displayed either in a detailed table or in a clear flow diagram.

5.1.1. LSC control panel

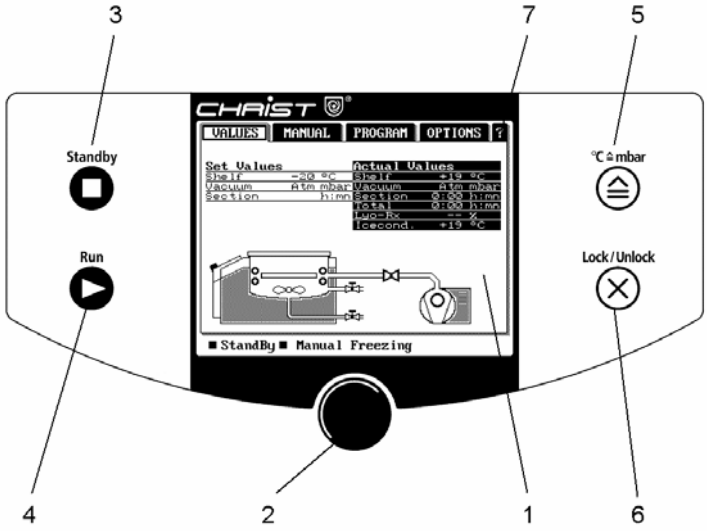


Fig. 1

1. ¼“ VGA LC display
2. Turn & push button ●
3. Function key “Standby” ■
4. Function key “Run” ▶
5. Function key “°C ≙ mbar” ⏏
6. Function key “Lock/Unlock” ⊗
7. Help function “?”

5.1.2. Brief description of operation

The LSC control system is operated via the central turn & push button ● and four function keys at both sides of the control panel ■ ▶ ≙ ⊗. The turn & push button is the main element for operating the control system. Turning the turn & push button ● positions the focus on the desired function in the menu or in the window. Pressing the turn & push button ● activates the selected function. Parameters (set values, etc.) are entered in the same way.

The following functions are assigned to the four function keys ■ ▶ ≙ ⊗:

Standby ■
: Switches the unit into the standby mode, i.e. all aggregates are switched off. This function is always available. It is usually used after the drying process is completed and before the manual aeration.

Run ▶
: Activates the phase that has been pre-selected in the “Manual” menu or starts a program. Phases are changed in the manual mode in the RUN state. This function is only available when all menus and windows are closed.

°C≙mbar ≙
: The conversion table “vapor pressure above ice“ for water is opened in another window. This function is only available in the mimic diagram, in the values window, in the programmer module or during the modification of a set value.

Lock/Unlock ⊗
: The function keys ■ ▶ ≙ and the turn & push button ● are locked against unintentional operation. The locking function is activated by pressing the key for about two seconds. The unlocking function is activated in the same way.

5.2. Visual components of the LSC control system

The LC display is divided into the three following areas:

- Menu bar
- Window area
- Status bar

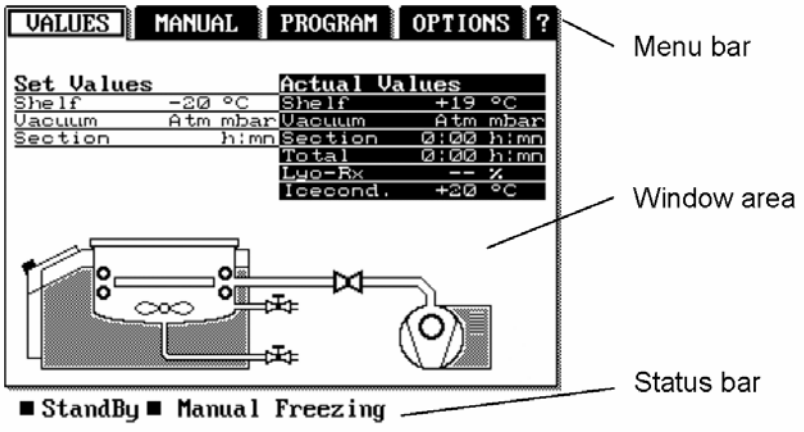


Fig. 2 Presentation on the LC display

5.2.1. **Menu bar**

The menu bar includes 5 menu tabs: VALUES, MANUAL, PROGRAM, OPTIONS, ? (Help). These menu tabs offer all configurations, parameters and operation modes of the unit.

The focus can be positioned on the desired tab by turning the turn & push button ◉ to the left or to the right.

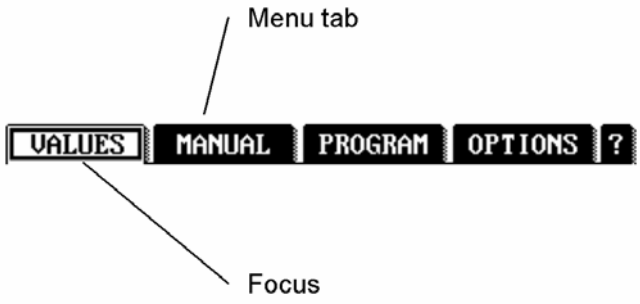


Fig. 3 Moving the focus in the menu bar

Press the button ⓿ to confirm the selected menu.

Fig. 4 Selected focus in the menu bar

The system opens a pop-up menu or a new window.

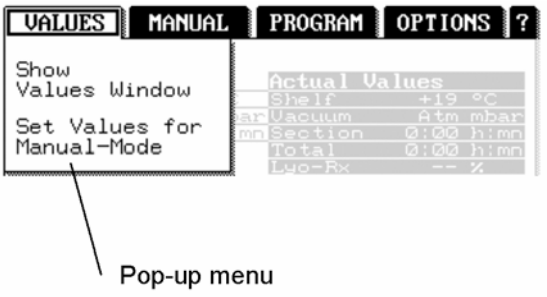


Fig. 5 Presentation of a pop-up menu

5.2.2. Window area

The main window covers the whole window area. Pop-up menus or dialog boxes are shown in front of the main window so that the main window is always in the background. Settings can only be modified in the active window in the foreground. This means that when a menu is open, the actual values in the main window cannot be modified.

Below is a description of the different window types of the LSC control system:

1. Pop-up menus
2. Dialog boxes
3. Main windows (covering the whole window area)
   a. Mimic diagram
   b. Values window

5.2.3. Pop-up menus

There are two different types of pop-up menus: Pop-up menus that are opened by selecting one of the main menu tabs (first-level pop-up) and second-level pop-ups that are opened by selecting a menu item from a first-level pop-up menu. The main menu tab is also used to close the menu of a first-level pop-up.

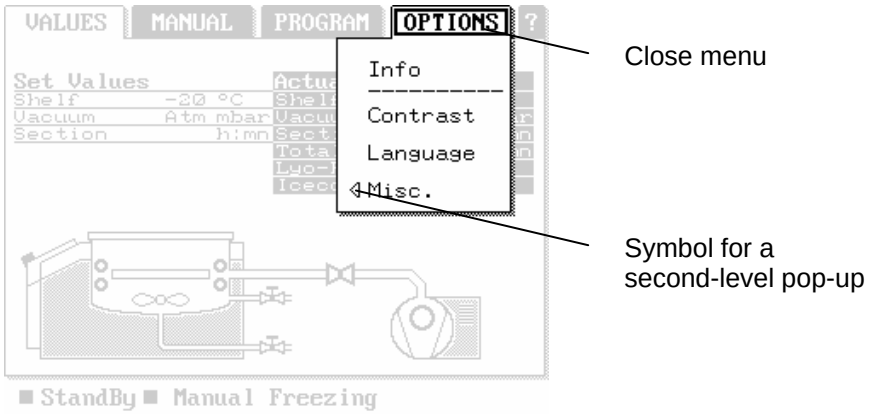


Fig. 6 First-level pop-up menu

In order to close a second-level pop-up menu, select the Close button which is available in all second-level menus. The second-level menu is closed and the system returns to the last menu item of the first-level pop-up menu.

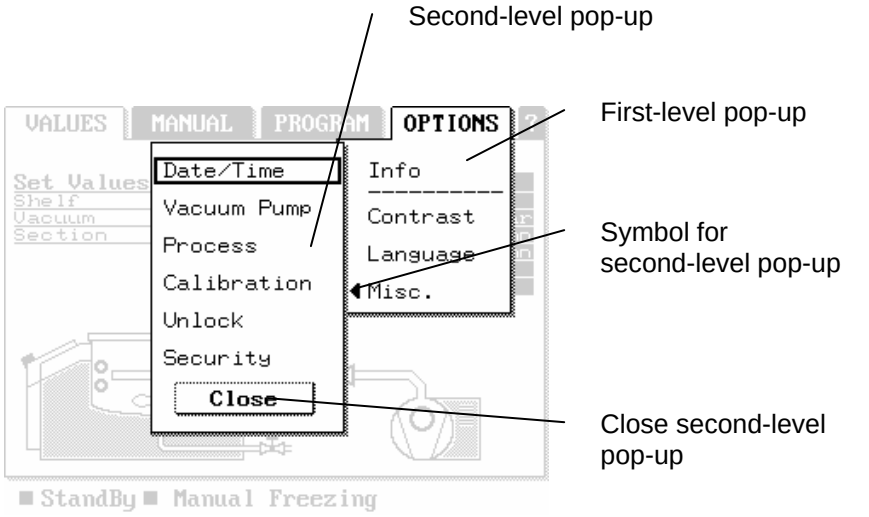


Fig. 7 First- and second-level pop-up menus

5.2.3.1. Dialog boxes

Dialog boxes are used to input parameters and to display additional information (e.g. vapor pressure curve above ice, set value changes etc.). A dialog box is usually opened by selecting a menu item or by pressing a function key. The process and

equipment information system is an exception since this dialog box is opened automatically by the system.

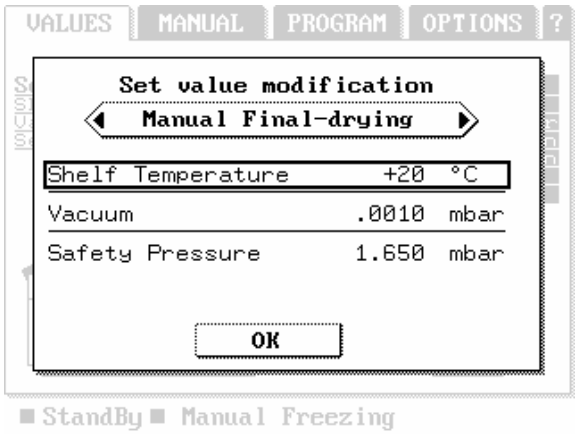


Fig. 8 Dialog box

The following control elements are included in the dialog boxes:

- Button:
  Buttons are used to confirm or to close a window.
- Scrollbar:
  The scrollbar is used to enter higher-order parameters.
- Radio button:
  A radio button is used to select a certain configuration.
- Parameter field:
  All other fields that can be focused are used to enter parameters, e.g. for set values, time constants, etc.

5.2.3.2. Main window

Mimic diagram

The mimic diagram shows the main process values, set values and actual values.

The set values always refer to the pre-selected or active phase (see status bar).

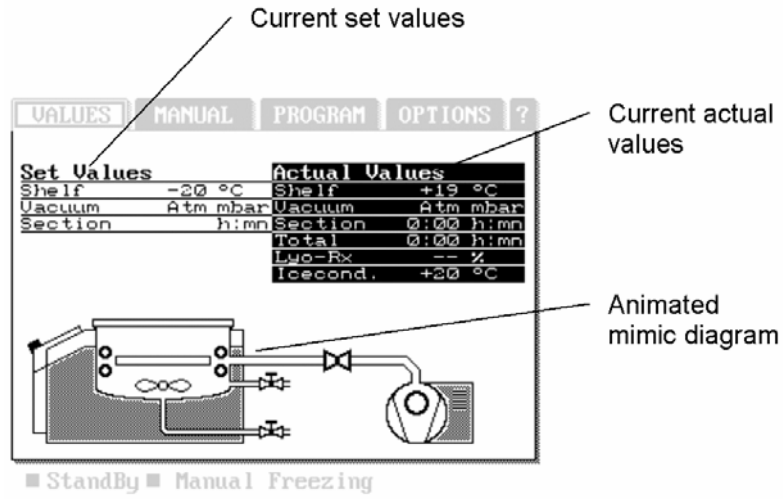


Fig. 9 Mimic diagram

The lower part of the window shows an animated mimic diagram with active components (symbols). The active components of the unit are the ice condenser, the vacuum pump, valves, the electrical heating unit for shelves and the fan in the ice condenser chamber. The symbols are shown completely filled-up during operation.

Anfang ALPHA1-4/2-4LSC

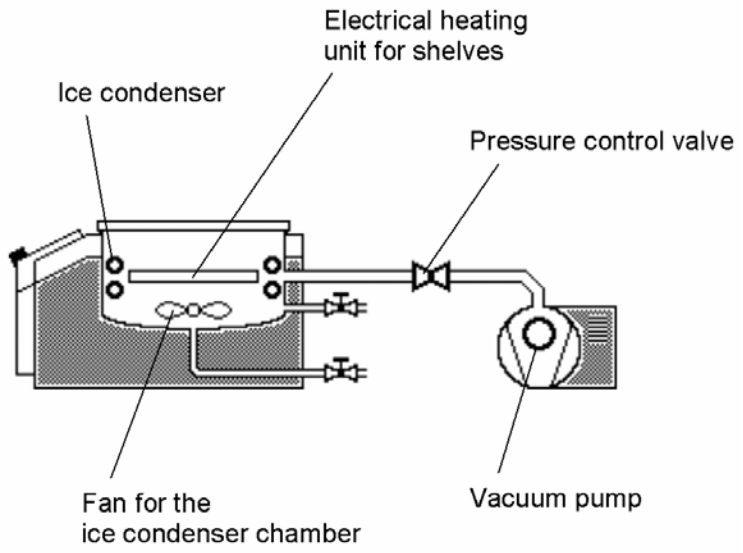


Fig. 10 Mimic diagram of ALPHA 1-4 LSC / ALPHA 2-4 LSC

Example for active components:

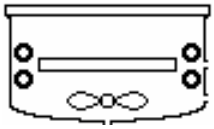


Fig. 11 Ice condenser and fan for ice condenser chamber are not active

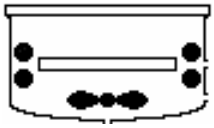


Fig. 12 Ice condenser and fan for ice condenser chamber are active

The mimic diagram is always shown after the unit has been switched on.

It is possible to jump from the mimic diagram to the values window by selecting the menu **VALUES** → `Show values window`.

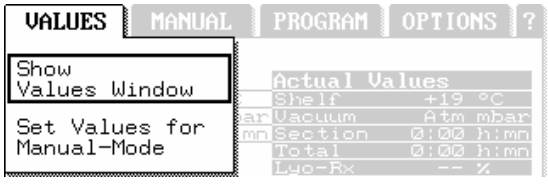


Fig. 13 Opening the values window

Values window

The values window compares set values and actual values of the running process in a clear manner.

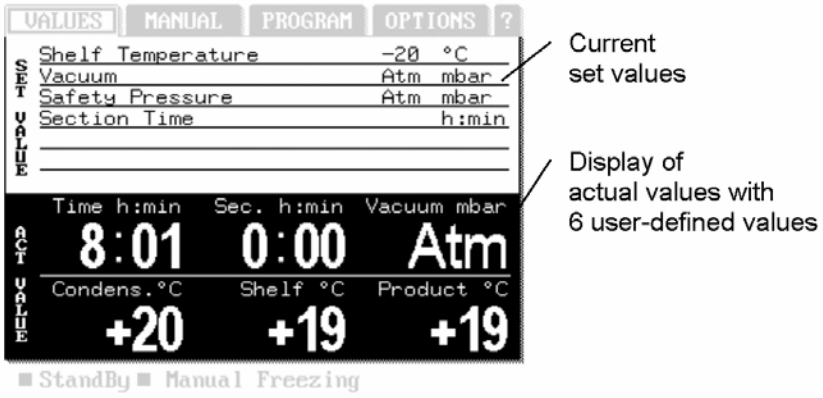


Fig. 14 Values window

The display of the actual values offers a special feature: The operator can select six actual values and assign them to the different display positions. In order to assign these six values select the menu **VALUES** → `Display configuration.`

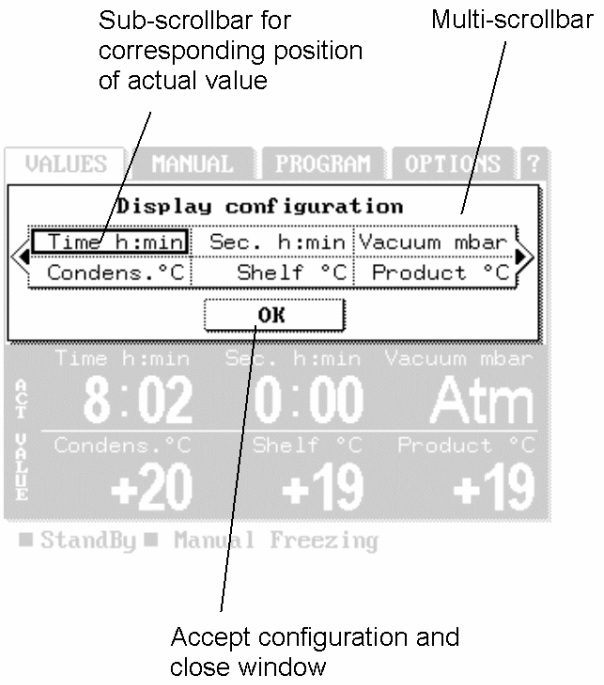


Fig. 15 Dialog box for the configuration of six positions for actual values

The 6 sub-scrollbars of the multi-scrollbar reflect the positions of the 6 actual values in the values window.

The following actual values can be selected via the sub-scrollbar:

- `Time h:min` (current time)
- `Tot. h:min` (total process time)
- `Sec. h:min` (time of process phase)
- `Vacuum mbar` (actual vacuum value)
- `Condens.°C` (ice condenser temperature)
- `Shelf °C` (shelf temperature)
- `Product °C` (product temperature)
- `Lyo Rx %` (optional)
- `Lyo Prod.°C` (optional)

In case a position should not present an actual value, select `--blank--` from the sub-scrollbar.

To switch from the values window to the mimic diagram select the menu item **VALUES** → `Show mimic diagram`.

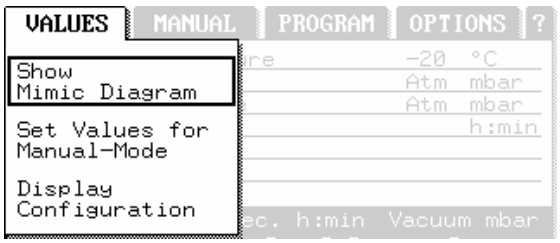


Fig. 16 Opening the mimic diagram

5.2.4. Status bar

The status bar always shows the current operating mode and the pre-selected or active phase. In the programmer mode, it also shows the current program section and the total program sections.

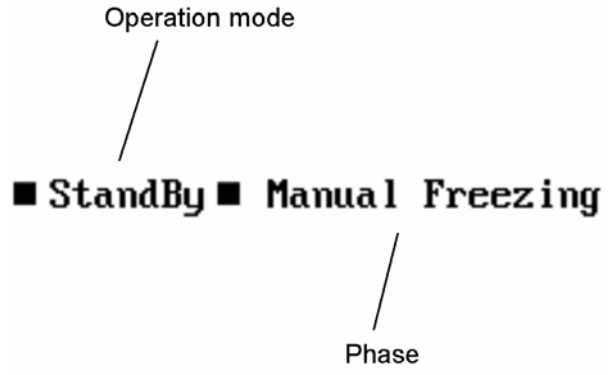


Fig. 17 Status bar in the manual mode

5.3. Function keys

Function key “Standby”

The function key “Standby” is used to switch off all aggregates. This function is always available (from every opened window). Usually, the drying process is finished with the “Standby” function before starting the manual aeration.

Function key “Run”

The freeze drying process is started with the “Run” key. In the manual mode, aggregates are switched on depending on the selected phase. If a program has been selected beforehand, it is started in the corresponding section. Phases are changed in the manual mode in the RUN state. The function “Run” is only available if all menus or windows are closed, i.e. the “Run” function can only be activated from the values window or the mimic diagram.

Please note that the aggregates are switched on by the control system one after the other with a short delay. Table 1 shows the delays for the units **ALPHA 1-4 LSC** and **ALPHA 2-4 LSC.**

	Time (sec.)	**Aggregate**
ALPHA 1-4	0	Refrigeration unit 1
	5	Vacuum pump
	10	All other aggregates
ALPHA 2-4	0	Refrigeration unit 1
	150	Refrigeration unit 2
	155	Vacuum pump
	160	All other aggregates

Table 1 Delay when switching on different aggregates

Function key “Lock/Unlock” ⊗

Press this function key ⊗ for 2 seconds to lock the unit against unintentional operation. That means that all inputs made with the turn & push button and with the three function keys are ignored by the control system. The following window is displayed instead.

Fig. 18 Dialog box "Device locked!"

The control panel can be unlocked by pressing the function key ⊗ again for 2 seconds.

Function key "°C≙mbar" ⏏

This function key can be used to convert the temperature of the ice condenser chamber or the vacuum in the drying chamber according to the vapor pressure curve above ice by means of a table. The conversion of the vapor pressure curve is necessary for a better orientation and a better understanding of the correlation between pressure and sample temperature during the freeze drying process (see chapter 2.1). Please note that the conversion table can only be opened if the mimic diagram, a values window or one of the set values windows is shown. With open pop-up menus or other dialog boxes, this function key is locked.

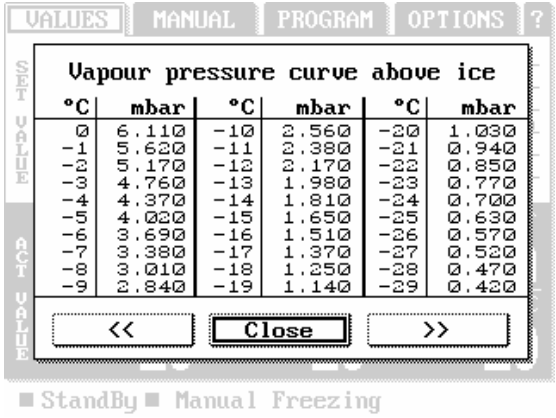


Fig. 19 Table for vapor pressure curve above ice

5.4. Manual mode

The manual mode enables the manual control of the process phases: Freezing, freezing + warm-up of the vacuum pump, main drying and final drying. In the manual mode, the set values for the different phases have to be entered before the process start. Then the phase has to be pre-selected. Press the function key "Run" ⏵ to start the process. When a phase is finished, the user has to

switch manually to the next phase without setting the unit to the standby mode. When all desired phases are finished – usually after the final drying phase – the process is finished by pressing the function key “Standby” . In the manual mode, the duration of the different phases is determined by the operator. It is not timer-controlled.

The following phases can be selected in the manual mode:

- `Freezing` (Only available with process A!)
- `Freezing + Warm-Up VP` (process A)
  `Cooling IC + Warm-Up VP` (process B)
- `Main-drying`
- `Final-drying`

The phase “Warm-Up VP” activates the vacuum pump with closed pressure control valve. This phase should last for at least 20 minutes to increase the service life of the pump.

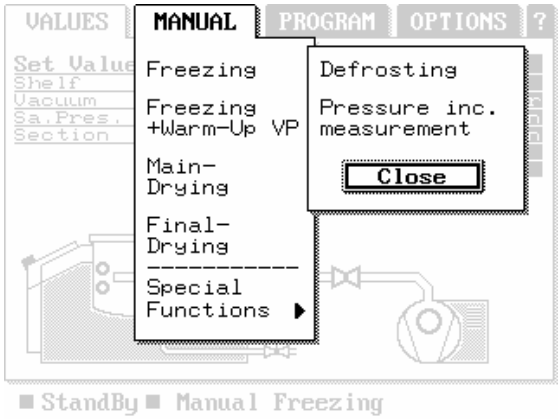


Fig. 20 Pop-up menu for manual control

Furthermore, the pop-up menu `Special functions` offers the following manual operations:

- Defrosting
- Pressure inc. measurement (pressure increase measurement).
- Preselection

5.4.1. Input of set values in the manual mode

The set values of the process are stored in a so-called nominal value set for each phase.

The dialog box for modifying set values can be opened via the menu **VALUES** → `Set Values for Manual Mode.`

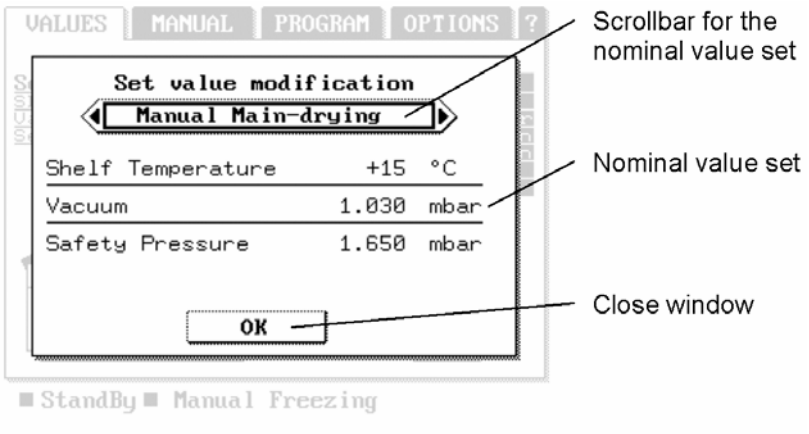


Fig. 21 Dialog box for the input of set values in the manual mode

To enter set values, select the corresponding nominal value set (phase) from the scrollbar. To do so, focus the scrollbar with (●) and confirm the selection with ●. The scrollbar is now in the selection mode, i.e. the desired nominal value set can be selected with (●). The corresponding current set values of this phase are shown. Press the confirmation key ● to exit the selection mode.

Fig. 22 Scrollbar in the selection mode

Now set the focus to the set value to be modified with (●) and confirm with ●. The editing mode is now active. The set value can be set in defined steps with the help of (●). Confirm with ● to exit the editing mode and to save the new set value.

Fig. 23 Editing mode for set values

5.4.2. Set values for manual freezing

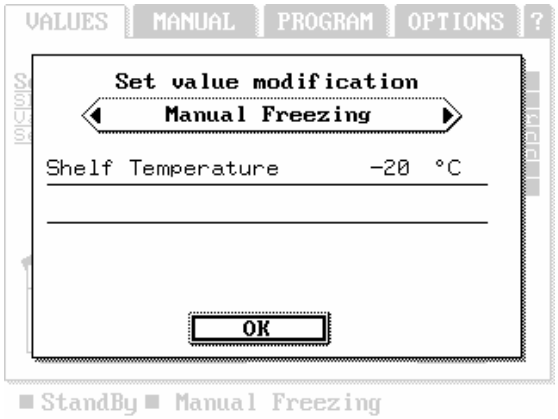


Fig. 24 Nominal value set for Freezing and Freezing + Warm-Up VP

Valid range of values:

Shelf temperature: -100°C ... +100°C

Max. range of values:

Shelf temperature: -100°C ... +100°C

5.4.3. Set values for manual main drying and final drying

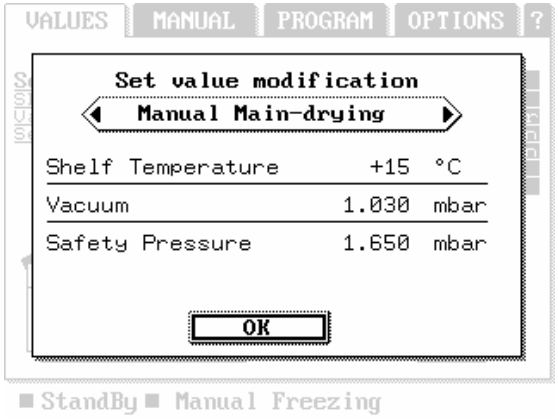


Fig. 25 Nominal value set for main drying and final drying

The set values “vacuum” and “safety pressure” are locked against each other. In this way the input of inadmissible nominal values is prevented. (Only a certain safety pressure can be selected. The corresponding temperature of the ice pressure curve of this pressure must be at least 5K higher than the one of the set value for the vacuum.)

Valid range of values:

Shelf:	-100°C .. +100°C
Vacuum:	0.0010 mbar ... safety pressure – 5°C
Safety pressure:	vacuum + 5°C ... 6.110 mbar and Atm

Max. range of values

Shelf:	-100°C ... +100°C
Vacuum:	0.0010mbar ... 4.020 mbar
Safety pressure:	0.0023mbar ... 6.110 mbar and Atm

The correlation between the vacuum and the temperature is shown in the conversion table “Vapor pressure curve above ice” in chapter 2.1.

5.4.4. Manual defrosting (option)

To defrost the ice condenser, select the phase “Defrosting” (`Manual → Special Functions → Defrosting`) and press “Run” ● to start the defrosting process. During the defrosting process, the ice condenser chamber is heated electrically by a heating collar. To finish the defrosting process, press “Standby” ■. If this button is not pressed, the defrosting process will end

automatically after the set defrosting time (see **VALUES** → `Set Values for Manual Mode`).

It is not possible to start the defrosting phase during an active phase (freezing, main drying, final drying). The unit has to be in the standby mode. During the defrosting process, it is not possible to select any of the other phases (freezing, main drying, final drying). In this case it is also necessary to switch the unit to the standby mode first. In addition, it is important that the chamber is completely vented, i.e. the actual vacuum value must be "Atm".

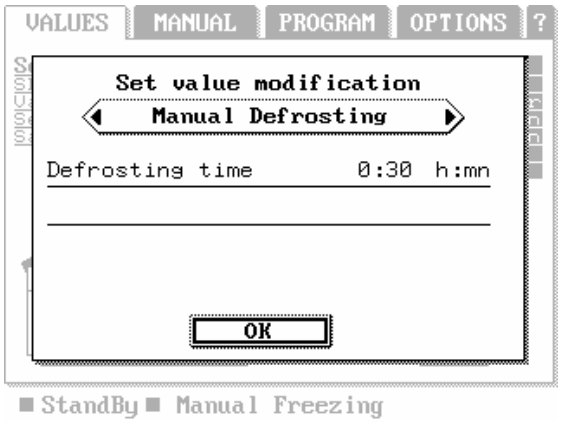


Fig. 26 Dialog box for manual defrosting

5.4.5. Manual pressure increase measurement (optional)

Select (`MANUAL → Special Functions → Pressure inc. measurement`) to activate the manual measurement of the pressure increase. This feature is only available in the “Run” ⏵ mode of the main drying phase or in the “Run” ⏵ mode of the final drying phase.

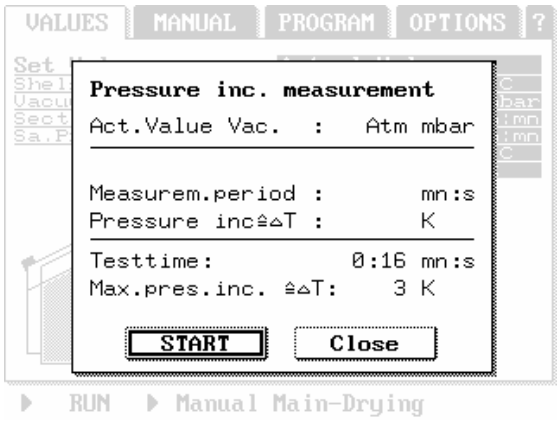


Fig. 27 Dialog box for the manual pressure increase measurement

Press START to start the pressure increase measurement. During the measurement, the intermediate valve prevents the flow of vapor from the drying chamber to the ice condenser. The pressure difference is displayed in accordance with the vapor pressure curve above ice.

The pressure difference is calculated as follows:

Pressure difference = actual vacuum value at the beginning of the measurement - current actual vacuum value

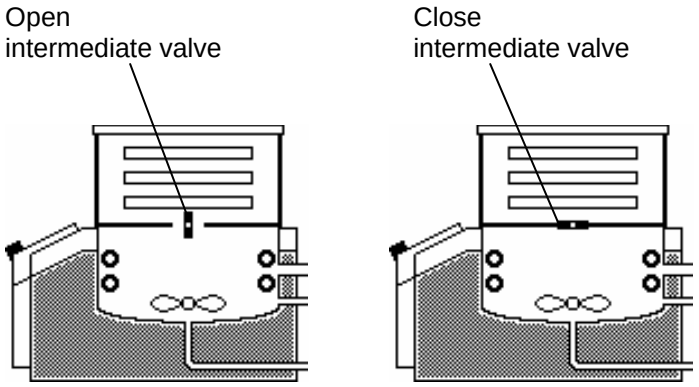


Fig.28 Intermediate valve between the drying chamber and the ice condenser

Press STOP to finish the pressure increase measurement. The values for the pressure difference and the measuring time are frozen. They are indicators for the current amount of water vapor and thus for the residual moisture content of the product.

The pressure increase measurement stops automatically when the test time is over or when the maximum permissible pressure increase is exceeded.

5.4.6. Preselection

Preselection for pressure control and air injection.
All preselections will be reactivated after:

- a phase change
- a program has been loaded
- a switch-over from “Run” to “Standby”

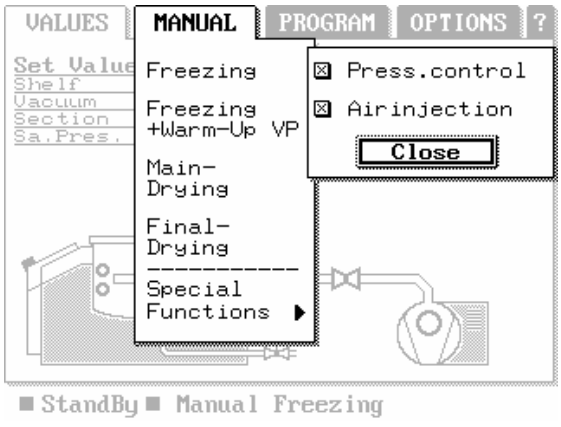


Fig. 29 Preselection menu

5.5. Programmer module (optional)

5.5.1. General information

The programmer module PGM-30 is used for a timer-controlled pre-programmed process (freeze-drying program).

A freeze-drying program is divided into several sections. Every section of the program has set values for the phase, the section time, the shelf temperature, the vacuum, the safety pressure and dT freezing/drying. The PGM-30 can store up to 30 individual programs. Up to 32 sections can be assigned to each program. A program has to comprise a minimum of 3 sections.

When the programmer module executes a freeze-drying program, it executes the created sections one after the other until the last section is completed. The defined set values for the shelf and the vacuum are node points that are used by the programmer module to calculate a linear function (ramp) with the corresponding gradient. This means that the set value does not change suddenly at the transition from one section to the next. Instead it has a steady ramp.

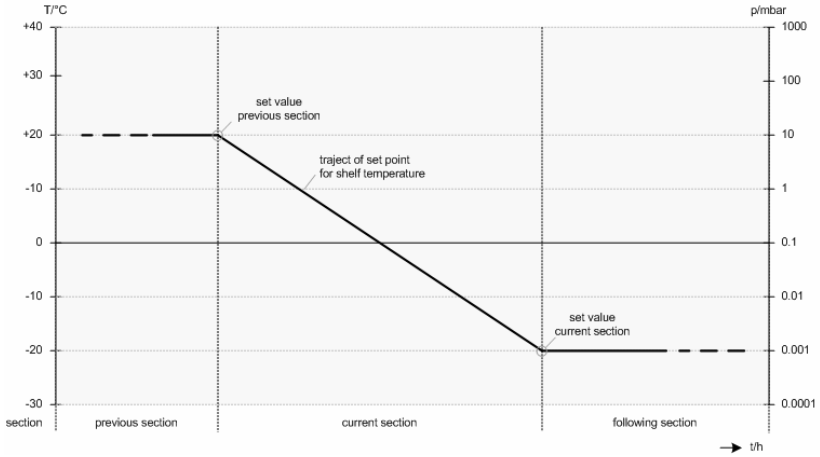


Fig. 30 The programmer module uses the section time and the set values of the current section and of the previous section and calculates a linear function (ramp) for the shelf temperature and the vacuum in the drying chamber.

The first section of a program is always “loading”. In this section, the start values for the calculation of the function and for the shelf temperature are defined. In general, the room temperature is stated. If the product is pre-frozen (process B, separate freezing and drying outside the ice condenser chamber), the corresponding product temperature should be stated. Section 2 and all other sections can be used for freezing, main drying and final drying, though it is not possible to switch phases. This means, for example, that the freezing phase cannot be selected after a main drying section.

The time for warming up the vacuum pump (warm-up phase) is automatically determined by the programmer module during the freezing phase. When the vacuum pump warms up, the freezing phase (section time) is not affected. The pressure control value is **closed** when the PGM-30 activates the vacuum pump during the

warm-up phase. As a result, the pressure inside the chamber is not affected.

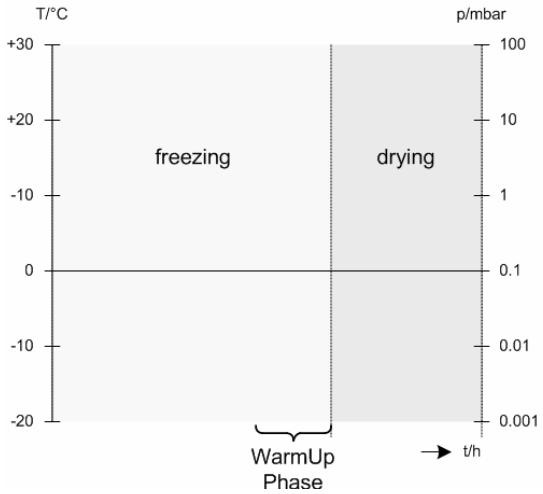


Fig. 31 The programmer module shifts the warm-up phase into the freezing phase so that it takes place prior to the first drying section

The programmer module data “set values”, “actual values” and “status” are shown at the usual locations in the values window, in the mimic diagram and in the status line.

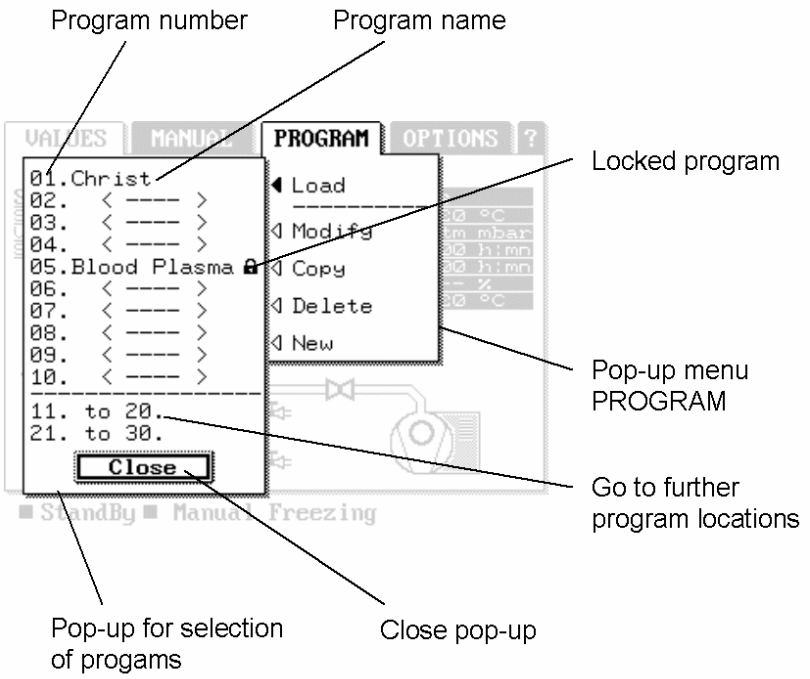


Fig. 32 Pop-up window of the programmer module

The programmer module can be activated by selecting the `Program` menu. This is only possible in case no program has been loaded so far. A pop-up menu opens and can be used to select the desired function (load, modify, copy, delete or new program).

5.5.2. Creating a new Program

In order to create a new program, select a free location under `Program` → `New`. Free program locations are marked by < `----` >. After the selection of a program location, the program editing mode is activated. As a program base, the standard program named “Christ” is created. The standard program can be customized by changing set values or by deleting or adding sections. The date of creation of the program is set to the current date.

The CHRIST standard program is a suggestion for drying aqueous solutions with a freezing point >-10°C in accordance with process A (freezing and drying on cooled shelves inside the ice condenser chamber).

	Set Values					
Section	Phase	Section Time [h:min]	Shelf Temperature [°C]	Vacuum [mbar]	Safety Pressure [mbar]	dT Freezing Drying [°C]
1	Loading		20			
2	Freezing	01:00	-20			OFF
3	Freezing	01:00	-20			OFF
4	Freezing	00:30	-15			OFF
5	Freezing	00:30	-15			OFF
6	Main Drying	00:10	-15	1,03	OFF	OFF
7	Main Drying	01:50	-15	1,03	1,65	OFF
8	Main Drying	01:00	-10	1,03	1,65	OFF
9	Main Drying	01:00	-10	1,03	1,65	OFF
10	Main Drying	02:30	20	1,03	1,65	OFF
11	Main Drying	09:30	20	1,03	1,65	OFF
12	Final Drying	00:10	20	0,001	1,65	OFF
13	Final Drying	00:50	30	0,001	1,65	OFF
14	Final Drying	02:00	30	0,001	1,65	OFF

Fig. 33 CHRIST standard program (table)

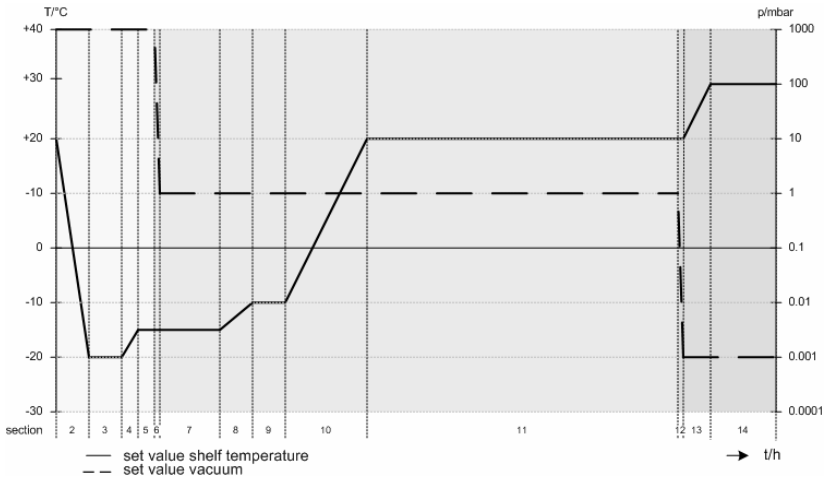


Fig. 34 CHRIST standard program (diagram)

5.5.3. Editing a program

An existing program can be adapted to your special freeze-drying process under **PROGRAM** → `Modify`. Select the program to be modified from the program list. If the program is locked for unauthorized persons, an additional four-digit user code has to be

entered. The user code is defined under **OPTIONS** → `Misc.` → `Security`.

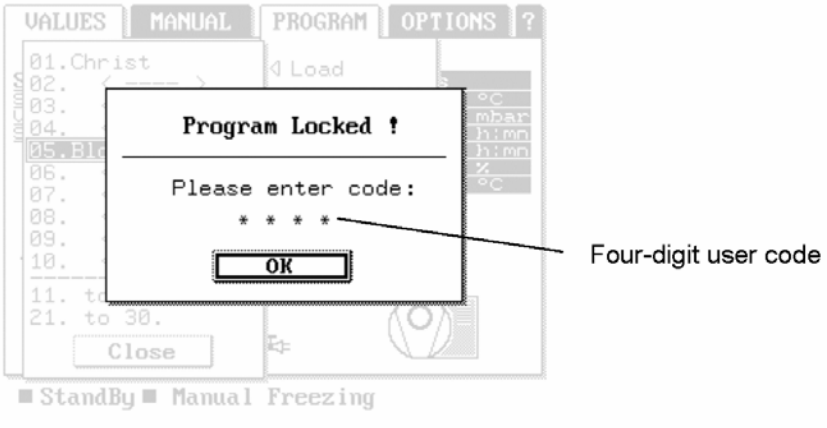


Fig. 35 Dialog box for entering the user code

The programmer module always shows 3 sections in order to make the connection with the previous and the section clear. The first section is always defined as “loading”. In this section, the start conditions of the program are defined. All other sections will be executed one after another. Please note that the order of phases of a program is always “loading”, “freezing”, “main drying” and “final drying”. It is not possible to switch phases. Example: You cannot have the “main drying” phase before the “freezing” phase.

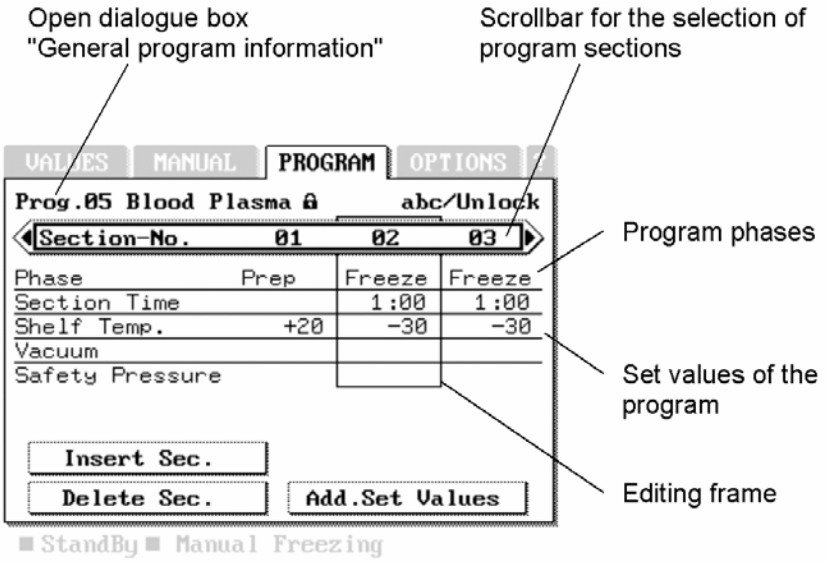


Fig. 36 The program editing mode

Select the **PROGRAM** menu to close the programmer module. This is necessary in order to save the modifications in the program memory.

Modifying a Program

The section to be modified has to be selected using the scrollbar. It is then displayed in the middle (marked by an editing frame). After exiting the scrollbar, the focus jumps to the editing frame. Now it is possible to modify the phases and set values. In order to

exit the editing frame, select the top focus position (section number).

Inserting a section Insert Sec.

This button inserts a new section at the section position in the editing frame. The set values are copied from the selected section. A maximum of 32 sections can be defined.

Deleting a section Delete Sec.

This button deletes a section at the section position in the editing frame. A program consists of at least 3 sections.

Additional set values Add.Set Values

- Warm-up vacuum pump
  Defines the time for the warm-up of the vacuum pump. For the warm-up, the vacuum pump is switched on automatically by the programmer module.
- Alarm LyoControl Rx
  The value “alarm LyoControl Rx” defines the minimum permissible value of the LyoRx sensor during the drying process. If the actual value falls below this minimum value, the shelf heating unit will be switched off. This prevents the product from thawing as a result of the excessive energy supply through the shelf temperature-control system. The LyoRx value is only evaluated during the main drying phase and during the final drying phase.
- dt shelf
  At the end of the section, the actual temperature of the shelf is compared to the set value. If the shelf temperature difference is beyond the permissible range (dt shelf), the section will be extended by the PGM-30 until the difference is in the permissible range. If the set value for "dt shelf" is set to OFF, the shelf temperature will not be checked at the end of the section.
- Pressure increase test during main drying
  See section 5.5.8 “Pressure increase test during main drying”
- Pressure increase test during final drying
  See section 5.5.8 “Pressure increase test during final drying”

5.5.3.1. General program information

The dialog box “General program information“ shows the program name and the locking state of the program.

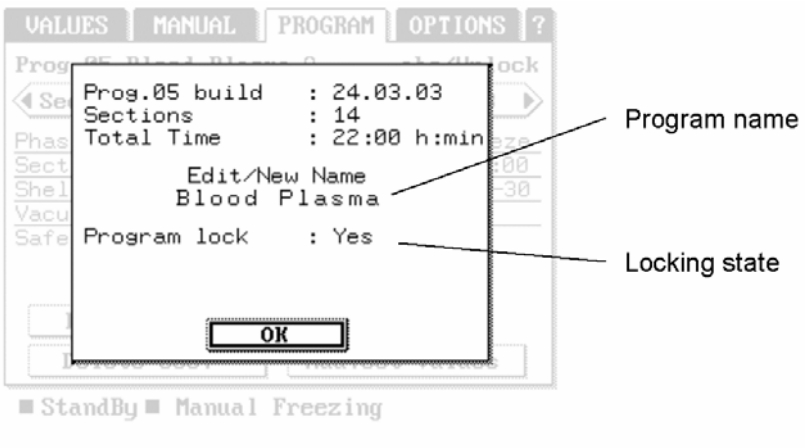


Fig. 37 General program information

5.5.4. Loading a program

Select the program under **PROGRAM** → Load. A dialog box opens showing general program information. The start section and the time for starting the section can be set as desired.

Press LOAD to load the program. The dialog box is closed and the status bar is updated. Press the function key "Run" ▶ to start the program.

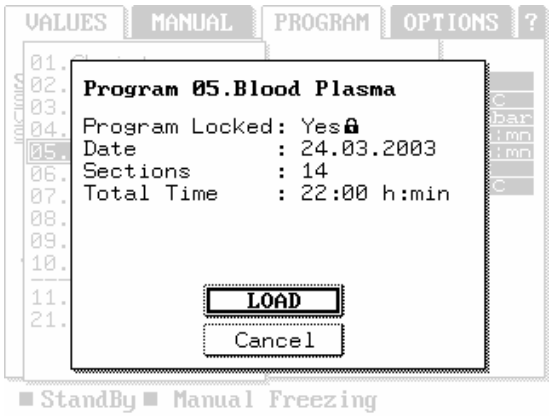


Fig. 38 Dialog box "Load program"

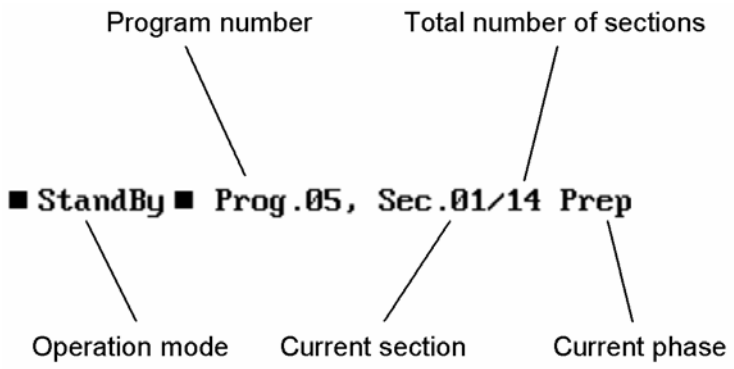


Fig. 39 Status bar in the programming mode

5.5.5. Copying a program

Select the program to be copied (source program location) under **PPROGRAM** → Copy. Only program locations that actually contain a program can be copied. If a program location is free, the selection will be ignored. If all 32 program locations already contain a program, the selection will be ignored, too. After the selection of the program, a dialog box opens showing general program information. The LSC control system suggests a free program location as the target program location. If necessary, this suggested location can be changed (through only free program locations can be selected). Press COPY to copy the source program to the target program location. The dialog box is closed. Select Cancel to exit the copying mode and to return to the program selection.

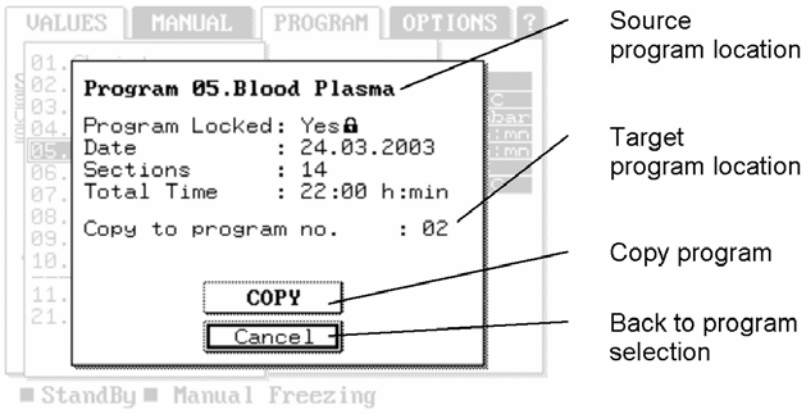


Fig. 40 Dialog box "Copy program"

5.5.6. Deleting a program

Select the program to be deleted under **PROGRAM** → Delete. Only program locations that actually contain a program and that are not locked can be deleted. In the case of free or locked program locations, the selection will be ignored. After the selection of the program, a confirmation dialog box opens. Press Delete to delete the program. The dialog box is closed. Press Cancel to return to the program selection.

Fig. 41 Dialog box "Delete program"

5.5.7. Delta t freezing/drying

Preselection of the maximum admissible difference between the shelf temperature and the product temperature at the end of a section. The unit only switches to the next process phase if the actual difference is below the set value.

If, for example, the shelf temperature during the freezing process is set to -40°C and a product temperature of at least -30°C has to be ensured before the “main drying” process can start, the admissible difference is +10K. In this case, enter “10” for the set value for delta t freezing.

If, for example, the shelf temperature during the drying process is set to +20°C and a product temperature of at least +15°C has to be ensured before the “final drying” process can start, the admissible difference is 5K.

If the difference is not reached within the preset section time, the section will continue until either the value is reached or the value falls below or exceeds the set value. A corresponding message is displayed in the process and equipment information system.

The temperature difference can be set in a range between 1 and 25. In order to ensure the beginning of the next process step, select a temperature difference that is not too small.

“delta t freezing” is measured by the temperature sensor “actual value product 1“. If the measurement is not desired or if it is not possible to use any product sensors, the measurement can be deactivated by selecting “OFF“ as the set value.

5.5.8. Pressure increase measurement during drying

In the program mode, the pressure increase measurement is always performed in the last drying phase (main drying or final drying). The next phase only starts after two successful pressure increase measurements in a row. In case there are no two successful measurements at the end of the section, a message is displayed in the process and equipment information system. The section is extended until two measurements are performed successfully.

Adapt the following parameters for the pressure increase measurement:

Max. Pres. Increase
The maximum admissible pressure increase for a successful measurement. The value is converted to K in accordance with the vapor pressure curve above ice.

Start pres. inc. test
Start of the first pressure increase measurement since the beginning of the last drying section.

Time between tests
Time difference between two pressure increase measurements.

Test time
During the test, the ice condenser is separated from the drying chamber. The pressure increase is analyzed after the test time.

If the pressure increase has to be measured during the execution of the program, the corresponding option checkbox must be selected.

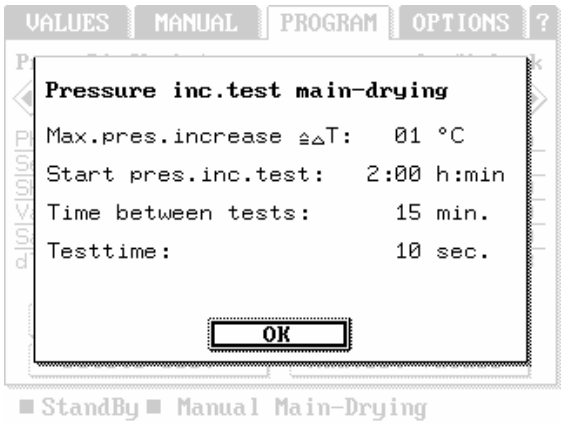


Fig.42 Parameters for the pressure increase measurement

5.6. Process and equipment information system

The process and equipment information system shows and saves malfunctions and messages. In this way, short-time defects or messages occurring overnight, for example, can be recognized the next day. Consequential malfunctions are also saved and displayed. The process and equipment information system is provided with a floating relay output for a central monitoring station.

The process and equipment information system distinguishes a message and a process or hardware error. In the event of an error or message, the process and equipment information system appears automatically on the screen. Simultaneously, a sound signal is given (5 times at 16-seconds intervals) until the error message is confirmed. In the event of process or hardware errors, the active operating lamp “Run” or “Standby” flashes. The information window cannot be closed until all errors and messages have been acknowledged.

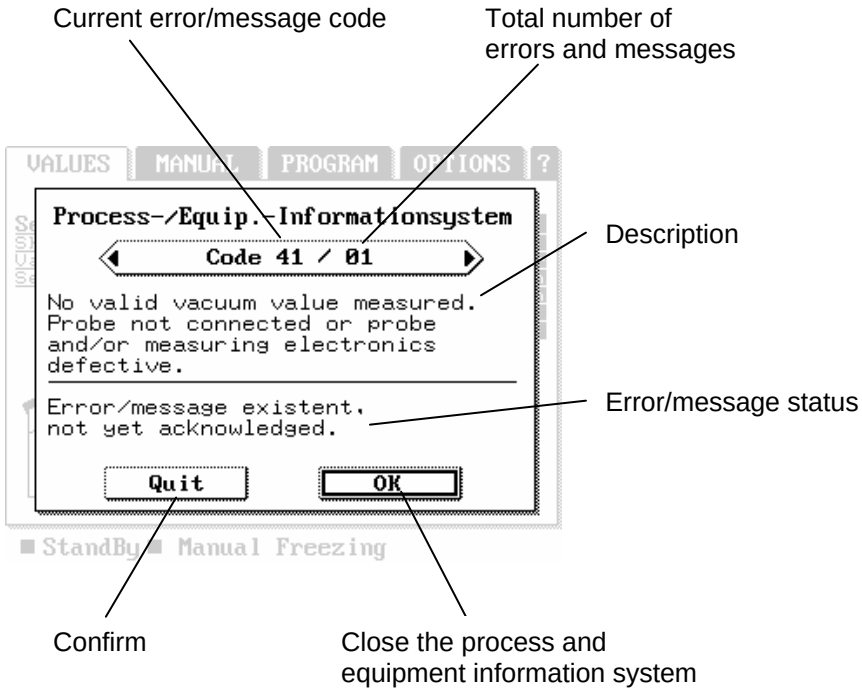


Fig. 43: Dialog box for the process and equipment information system

The following messages are displayed in the process and equipment information system:

5.6.1. Process messages

For messages referring to hardware errors please see chapter 6.7.1.

Code 03

`Safety pressure was fall below and exceeded again during the drying process.`

The error message is displayed if the pressure has fallen below the preset safety pressure and has then exceeded it. Please check whether the preset value for the safety pressure is too low or the preset value for the quantity of the sample to be dried or the shelf temperature are too high. In this case, the temperature of the ice condenser may rise too high because of the high load.

Code 20

`Alarm Lyo Control Rx`

LyoRx value is out of range. Shelf heating is turned off.

Code 21

`Product to wet. Section was extended`

This error message is displayed if the program fails to perform two successful pressure increase measurements in a row at the end of a section. The section is extended.

Code 22

`Note: dT Freezing/Drying`
`Producttemp.1 ist out of limit at section end.`

See also chapter 5.5.7 “Delta t freezing/drying”

Code 24

`Maintenance interval reached. Perform maintenance or contact local service.`

See also section 5.7.5 “Maintenance of the freeze dryer”

Code 25

`Shelf temperature has not been reached at section end.`

Only in the programmer module! This message is displayed if the shelf temperature differs in an inadmissible way from the set value for the shelf temperature at the end of a section.

Code 26

`Program finished.`

Only in the programmer module! The program run has been completed.

Code 29

`Maintenance of the vaccum pump is necessary. Please maintenance the vacuum pump and reset the working hour meter.`

It is possible to set a maintenance interval for the vacuum pump under **`OPTIONS`** → `Misc.` → `Vacuum Pump` (see “Options”). The message is displayed in the process and equipment information system when maintenance is due. The message has to be confirmed. You should now perform the required maintenance and then reset the operating hour counter under **`OPTIONS`** → `Misc.` → `Vacuum Pump`.

Code 43

Water in ice condenser chamber

There is still water in the ice condenser chamber.

5.7. Options

The **OPTIONS** menu is used to enter elementary configurations of the control system.

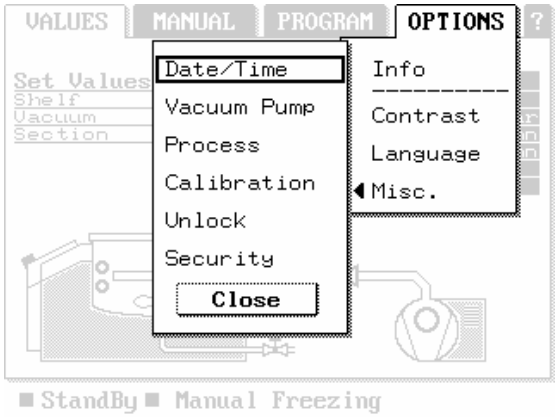


Fig. 44 Options menu

5.7.1. Info

Select the menu item **Info** to open the process and equipment information system. Saved messages or errors can be read here.

5.7.2. Display contrast

The contrast of the LC display has to be modified depending on the lighting conditions and the visual angle. Open the corresponding dialog box under **OPTIONS** → Contrast. Position the focus on the scrollbar with (●) and ⓓ, i.e. adjust the optimum contrast with (●) and confirm it with ⓓ. The value range for the contrast is restricted so that even with 0 % or 100 % the content of the LC display can still be seen from a certain visual angle.

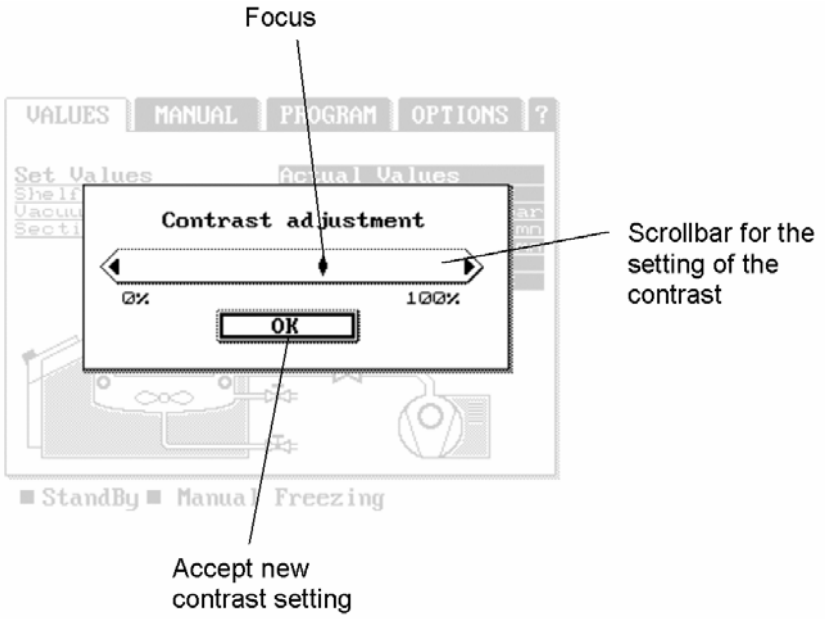


Fig. 45 Dialog box for setting the contrast

5.7.3. Language

To set a language, select **OPTIONS** → Language. Turn the turn & push button to select the corresponding flag and confirm it with . Afterwards the mimic diagram is shown using the new language.

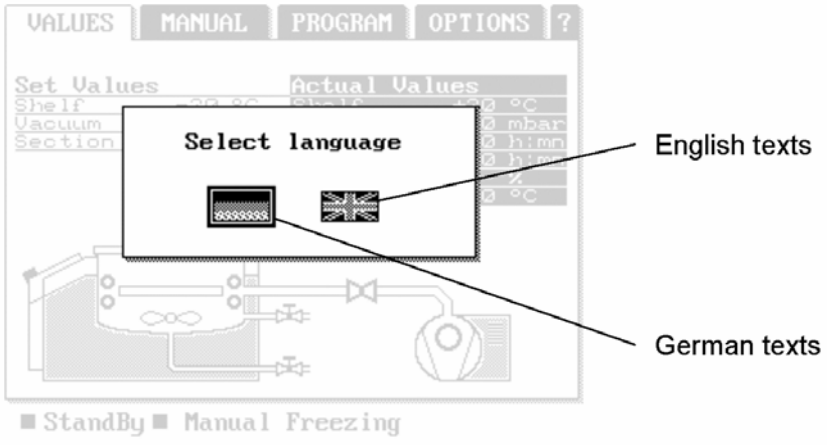


Fig. 46 Dialog box for selecting the language

5.7.4. Changing the date and time

The LSC control system is equipped with a built-in clock with a buffer battery. The clock has to be reset after a failure of this buffer battery. The time can be set under **OPTIONS** → Misc. → Date/Time. The date and time that are currently set are used as set values when the window is opened.

To set the date, proceed as follows:

Set the current values for day, month and year und confirm these settings with SET.

To set the time, proceed as follows:

Set the current values for hour and the next minute. When the minute changes, confirm the new setting with SET. The counter of the seconds will be reset to zero.

Example:

The current time is 10:23:15 [hour:min:sec]. Hence the time to be set is 10:24 [hour:min]. Confirm the new setting by pressing SET when the minute changes to 10:24:00 [hour:min:sec].

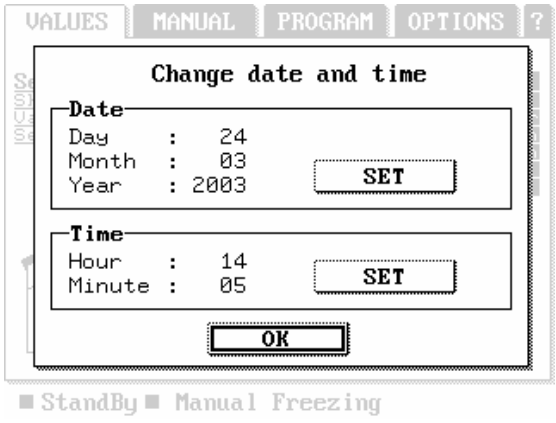


Fig. 47 Dialog box for date and time

5.7.5. Maintenance of the freeze dryer

Maintenance of LSC freeze dryers is due after a maximum of 3000 operating hours or after one year after the last maintenance. When maintenance is due, message code 24 is displayed in the process and equipment information system. After the maintenance, reset the counter under **OPTIONS** → Misc. → Maintenance by pressing the RESET button. The dialog box also shows the current number of operating hours since the last maintenance and the date of the last maintenance in the format [month.year].

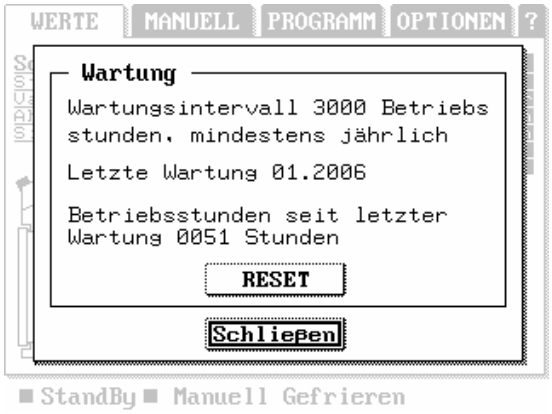


Fig. 48 Dialog box showing the maintenance intervals of the freeze dryer

5.7.6. Maintenance intervals of the vacuum pump

The maintenance intervals of the vacuum pump can be defined and reset under **OPTIONS** → `Misc.` → `Vacuum Pump.`

The following maintenance intervals can be selected: 100, 200, 300, 400, 500, 1000, 2000, 3000, 4000 hours. If the interval is set to ∞ hours, the system will never display any maintenance messages.

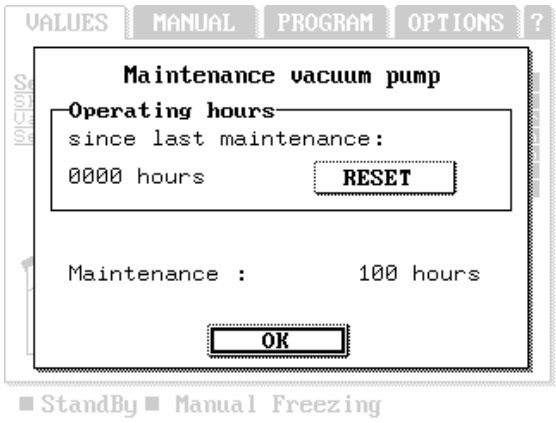


Fig. 49 Dialog box showing the maintenance intervals of the vacuum pump

5.7.7. Process

This dialog box can be opened under **OPTIONS** → `Misc.` → `Process`. It is used to preset the type of process (see chapter 3). The mimic diagram of the unit changes accordingly. If process B is selected, the phase `Manual freezing` is no longer available since the product is frozen outside the unit (e.g. in a deep-freeze). The phase `Manual cooling IC + warm-up VP` is still available in process B since it is used mainly to warm-up the vacuum pump and to precool the ice condenser.

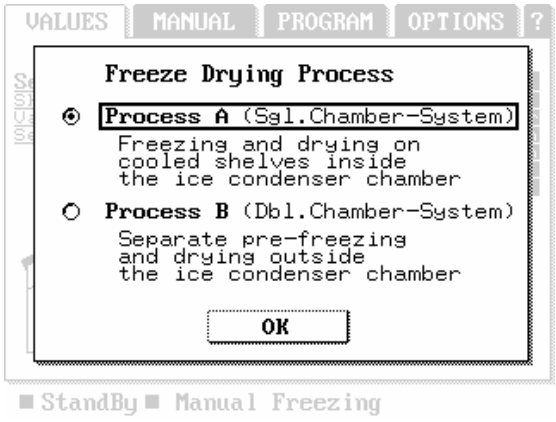


Fig. 50 Dialog box for selecting a process

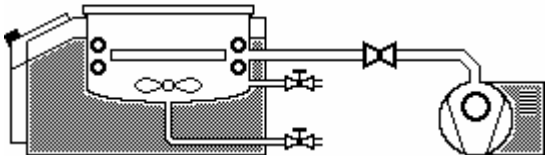


Fig. 51 Mimic diagram process A

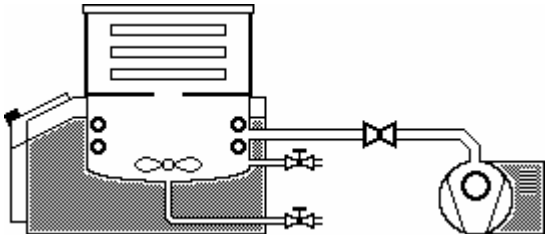


Fig. 52 Mimic diagram process B

5.7.8. Unlocking

This dialog box can be opened under **OPTIONS** → `Misc.` → `Unlock`. A six-digit keyword has to be entered.

Fig. 53 Dialog box for unlocking

There will be a long sound signal (about 1 s) in case an invalid keyword is entered. If the keyword is accepted, there will be a short sound signal.

5.7.9. Security

The menu item **OPTIONS** → `Misc.` → `Security` is used to define a four-digit user code to protect the program data against unauthorized access. First, a dialog box opens. It is used to enter the current user code. A new code can only be defined after this current code has been entered correctly. The new code is valid for all program data!

In the supply state, the user code is "ØØØØ".

Fig. 54 Dialog box for entering the old user code

There will be a long sound signal (about 1 s) if a wrong user code is entered. If the user code is accepted, there will be a short sound signal.

Fig. 55 Dialog box for the entering the new user code

5.7.10. System calibration

A dialog box showing all the values that are necessary for a calibration can be opened under **OPTIONS** → `Misc.` → `Calibration`.

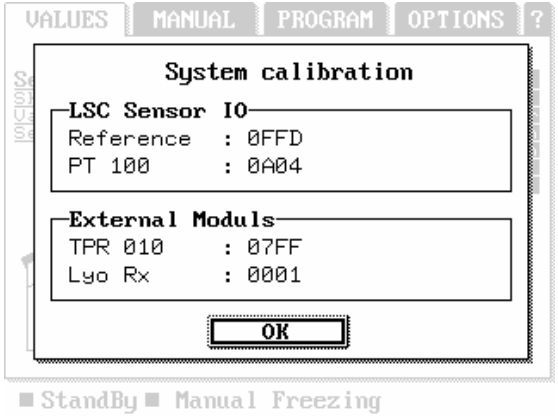


Fig. 56 Dialog box for calibrating the system

Two calibrations are possible: the calibration of the IO sensor card and the calibration of external measuring modules (Pirani and Lyo Control).

LSC Sensor IO

The LSC sensor IO card processes analog measuring signals such that they can be processed by the control system in the form of digital signals. The calibration is performed once at the factory.

PT 100 temperature sensors

In order to adjust the PT100 sensors of the freeze dryer, the "LSC Temperature Adjustment Tool" (part no. 135504) is required.

5.8. Integrated brief description

The integrated brief description does not replace a complete operating manual, but it can give an overview about the operation, parameterization and operating modes of the unit.

The brief description is opened by selecting **?** in the menu bar. The system then shows a list of topics. Use the buttons << and >> to browse the contents page by page. Use the Close button to exit the brief description and to go back to the main window.

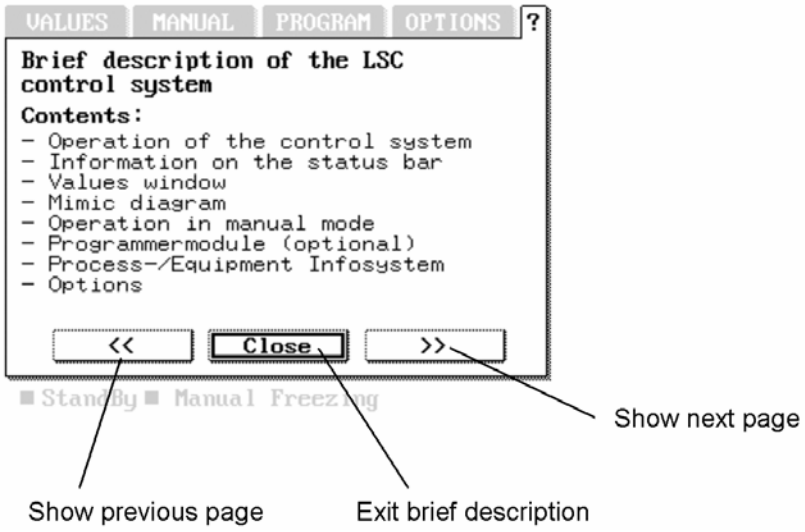


Fig. 57 Contents of the brief description

6. Troubleshooting

6.1. Power failure

The control system continues with the process after a power failure. The pre-selected conditions remain saved even during an automatic process.

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 when the power failure occurred.

It is important to distinguish between the main drying phase and the final drying phase:

The product is in the final drying phase when the residual moisture has reached approx. 5 %. Below this value, the product is generally not damaged by a power failure.

If the product is in the main drying phase, we recommend aerating the unit, taking out the product and storing it in a deep-freeze. The defrosted condensate must be drained off prior to the next start.

6.2. Insufficient vacuum

Special attention must be paid to the high-vacuum valves. The drain valve, the aeration valve as well as the rubber valves must be checked.

After disconnection of the mains plug, we recommend removing the side panels of the unit, disconnecting the hose inside the unit from the drain valve, sealing the hose end with a rubber stopper and evacuating the unit. If the unit now reaches the necessary operating pressure, there is a leak in the valve. This may be caused by residues from the drying process, fluff from cleaning cloths or wear of the O-rings of the valve.

To eliminate this problem, we recommend evacuating and aerating the unit through the drain valve so that any particles that the unit is evacuated and vented by means of the drain valve so that any deposits are removed. If necessary, the O-rings must be replaced.

If the leak cannot be eliminated in this way, the drain valve must be cleaned or replaced.

Please proceed in the same way in case of a leak in the aeration valve.

Check the lid seal for dirt or damage. If necessary, it must be cleaned or replaced.

Ensure that the entire surface of the ground-in stopper of the attachable drying chamber is greased with vacuum grease.

When using drying chambers with several connections for rubber valves, the valves should be removed and the connections should be sealed with rubber stoppers. To check the valves, they are connected one after the other and tested under vacuum.

Check the vacuum sensor for soiling, e.g. water residues.

The vacuum sensor has a limited life time due to its design principle. Vacuum sensors are available as spare parts (VSP62, part no. 125468).

Check the vacuum display with a suitable testing unit (if available).

In order to locate a possible leak, we recommend that the vacuum measuring sensor is directly connected to the inlet side of the vacuum pump. If the warm vacuum pump reaches a final pressure of at least 0.011 mbar, the vacuum pump and the measuring system can be considered as being in working order. There is probably a leak in the unit if the insufficient vacuum is not caused by an insufficient ice condenser temperature.

Check the oil level in the vacuum pump, change the oil if it is contaminated and carry out pressure test.

If the above-mentioned steps show no result, the next step is to check all of the small flange connections (see chapter 4.8.2) and especially the position of the centering rings. It is usually not necessary to grease the sealing rings with high-vacuum grease.

The vacuum pump does not switch on:

The vacuum pump is equipped with a protective switch for the drive motor.

(Please refer to the separate operating manual of the vacuum pump.)

General information:

The vacuum checks should be carried out when the ice condenser is deep-frozen.

6.3. Unit does not work

If the display does not work after the mains switch has been actuated, the following tests must be performed:

Is the mains switch plugged in?

Check the on-site fuses.

Check the automatic circuit breaker 1F1 at the back panel of the unit.

Check the internal fuse 202F3.

6.4. Heating unit is not activated

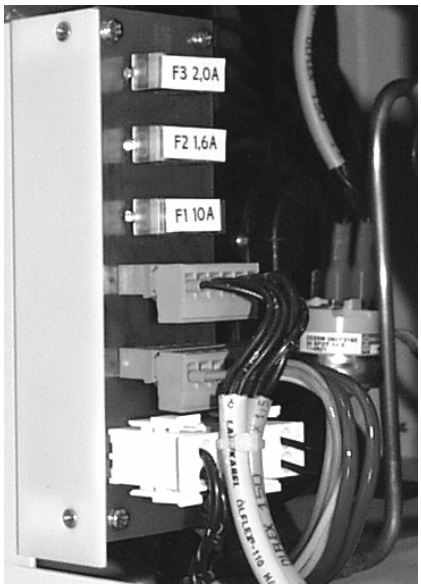
Please note that during the main drying phase and during the final drying phase the heating unit is not activated until the preset safety pressure is reached.

If the heating unit is not activated even though the pressure is below the safety pressure, please check the fuse 202F1.

6.5. Fan (ice condenser chamber) is not activated

Check the precision connection in the ice condenser chamber.

Check the internal fuse 202F2.



Internal fuses

The internal fuses 202F1, 202F2 und 202F3 are located behind the left side panel. Switch the unit off and disconnect the mains plug. Remove the left side panel of the unit.

6.6. Insufficient ice condenser temperature or shelf temperature

The refrigeration unit is equipped with a protective device against overpressure in the refrigeration system and with a thermal motor protection switch.

The protective devices trip

when the ambient temperature is too high,

when the air circulation of the heat exchanger of the refrigeration system is insufficient,

when the refrigeration system is overloaded.

The refrigeration unit is automatically switched off in these cases.

Once the permissible operating conditions are reached again (after a few minutes), the refrigeration unit is switched on automatically via the motor protection switch or via the pressure switch.

Important errors are indicated by the process and equipment information system.

The minimum ice condenser temperature of approx. -55°C or -85°C is reached when the ice condenser is not loaded and the ice condenser chamber is evacuated.

Sufficient air circulation is very important. Do not place any objects behind the unit!

6.7. Process and equipment information system

Process messages see chapter 5.6.1.

6.7.1. Error messages

Code 01

`Vacuum of 6.11 mbar has not been reached within 15 minutes. Vacuum pump was switched off. Vacuum leakage or pump defective.`

This error occurs when the pressure in the ice condenser chamber has not reached 6.11 mbar after 15 minutes of operation with open pressure control valve. There must be a vacuum leak in the system. The pump is switched off to avoid it pumping against the atmospheric pressure. The cause may be a vacuum leak (aeration valve or drain valve not closed, lid or drying chamber not mounted in a vacuum-tight way, missing ground-in stopper in the lid or in the drying chamber) or a defect of the vacuum pump (check whether the mains switch of the vacuum pump is switched on). The pressure control valve is closed and the vacuum pump is switched off. Please eliminate the error and confirm the message afterwards.

Code 02

`Safety pressure has not been reached within 20 minutes.`

This error occurs when the preset safety pressure is not reached within 20 minutes after the start of the vacuum pump (with open pressure control valve). Please check whether the value set for the safety pressure is too low.

Code 03

See chapter 5.6.1.

Code 04

`Excess temperature “ice condenser”. Temperature “ice condenser” is 65°C or more! Freeze Dryer was switched into Standby.`

This error occurs when the temperature of the ice condenser exceeds +65°C. At the same time, all active units are switched off, i.e. the system is automatically switched to standby mode. The error message may occur when the temperature of the ice condenser exceeds +65°C when defrosting the system. The refrigeration unit is switched off in order to avoid an operation or a start at too high temperatures. The unit cannot be reactivated until the ice condenser temperature is lower than +65°C and after confirmation of the error message.

Code 21

See chapter 5.6.1.

Code 22

See chapter 5.6.1.

Code 23

`AC power failure`

A power failure is detected if the voltage supply is interrupted under operating conditions (running mode). The process is resumed when the voltage supply is available again.

Code 24

See chapter 5.6.1.

Code 25

See chapter 5.6.1.

Code 26

See chapter 5.6.1.

Code 27

`Note: Invalid Control System`

Please contact our service department!

Code 28

`Note: Invalid System Module`

Please contact our service department!

Code 29

See chapter 5.6.1.

Code 30

`Note: Error in configuration memory. Please verify user defined parameters and check the back-up battery.`

This error message is displayed when the system is switched on and the buffer battery power for the settings and the real-time clock is too low. Please replace the buffer battery. Then it is necessary to reset the clock (see chapter 5.7.4). The settings (setpoints, etc.) - except for the modifications made in the last operating phase - are automatically reloaded from the second read-only memory so that they are available even after a failure of the buffer battery. Nevertheless, it is recommended to check the settings.

Code 31

`Note: Error in configuration memory. Factory settings were loaded! Please check the back-up battery.`

Please contact our service department!

Code 32

`Overpressure refrigerator RM1.`

Overpressure in the refrigeration unit RM1, released via the pressure switch. Please check the ambient temperature, the air circulation of the heat exchanger of the refrigeration unit and the load of the unit.

Code 33

`Overpressure refrigerator RM2.`

Overpressure in the refrigeration unit RM2, released via the pressure switch. Please check the ambient temperature, the air circulation of the heat exchanger of the refrigeration unit and the load of the unit.

Code 34

`Overpressure refrigerator RM3.`

Overpressure in the refrigeration unit RM3, released via the pressure switch. Please check the ambient temperature, the air circulation of the heat exchanger of the refrigeration unit and the load of the unit.

Code 36

AC power failure L2

Power failure in phase L2. Please check the on-site fuses.

Code 37

AC power failure L3

Power failure in phase L3. Please check the on-site fuses.

Code 38

Excess temperature liquid for temperature control shelves

The temperature-control liquid has exceeded the upper limit. The heating unit for the temperature-control liquid is switched off. The heating unit is not reactivated until the temperature-control liquid has fallen below a lower limit. The limits are set in the thermostat.

Code 39

Level liquid for temperature control fall below

There is not enough temperature-control liquid in the brine compensation reservoir.

Code 40

Temperature sensor “ice condenser” defective or not connected.

This error occurs when the temperature sensor is not connected properly or if it is damaged. Please contact our service department.

Code 41

No valid vacuum value measured. Probe not connected or probe and/or measuring electronics defective.

This error occurs when the heating wire of the vacuum sensor is damaged, the electronic measuring value device is defective or the vacuum sensor is not connected properly.

Code 43

See chapter 5.6.1.

Code 44

Temperature sensor shelf heating defective

This error message is displayed, if the temperature sensor is either not connected or defective. Please call customer service.

7. Maintenance

7.1. 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.

7.2. Heat exchanger

A laminated 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 soiling of the heat exchanger leads to a decrease in performance and may cause a failure of the unit!

Please note: The unit must not be turned upside down!

7.3. Calibration and maintenance of the vacuum sensor VSP62

7.3.1. Maintenance of the vacuum sensor:

The service life of the tungsten wire is limited due to the chemical reaction with the components of the surrounding gas. Tungsten reacts with carbon and forms tungsten carbide. As a result, the wire gets brittle and breaks. The service life of PIRANI measuring tubes can only be increased if they do **not** get into contact with carbon-containing substances.

A freeze dryer should be aerated and the dry product should be removed at the end of the drying process. Then the system should be defrosted with warm water (+50°C max.) while the drain valve is open. Then the drying chamber has to be wiped dry.

Do **not** simply let the freeze dryer stand at room temperature for a long period of time since this is the time during which the tungsten wire reacts with the surrounding atmosphere!

In addition, it is recommended to evacuate the unit for approx. 30 minutes with an **open** gas ballast valve after defrosting and drying in order to remove any residues which might react with the tungsten wire.

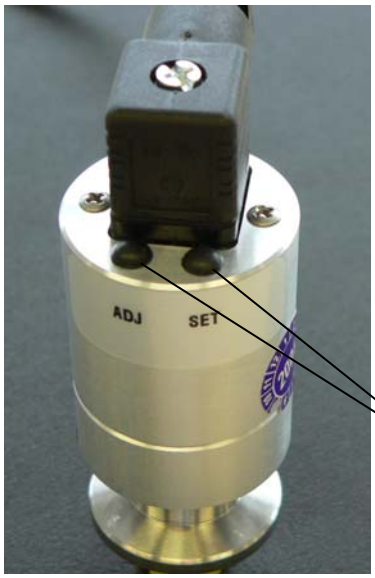
7.3.2. Replacement and calibration

The vacuum sensor has to be replaced if "---" is shown in the vacuum field after the aeration of the system instead of "Atm" and if the process and equipment information window shows error message 41. If after the warm-up phase and the aeration of the unit the vacuum display still does not show "Atm" but a value like "800", "600" or "400" mbar, for example, the vacuum sensor has to be recalibrated. The warm-up phase of the vacuum measuring sensor takes at least 5 minutes.

Calibration of the vacuum sensor:

The vacuum sensor has two calibration buttons labeled "adj" and "set". The buttons are protected by protective caps. Remove the protective caps of the buttons prior to calibrating the sensor. Use a small pen (max. ∅ 2.5mm) to actuate the buttons. Refit the protective caps after the calibration.

The digital recalibration to atmospheric pressure (Atm) and zero is possible directly at the vacuum sensor using the button labeled "adj" (adjust). The measuring transducer automatically recognizes the calibration point. If a zero calibration has to be performed, the actual pressure should be lower than 5.0×10^{-5} mbar (factory setting). Alternatively, the sensor can also be calibrated to a reference value (see below). We recommend a warm-up phase of at least 5 minutes before the calibration.



Buttons with protective caps for calibrating the vacuum sensor

Calibration to a specific zero reference value:

To perform the calibration, the actual pressure must be lower than $1.0x10^{-2}$ mbar. After pressing of the button labeled “set”, the actual vacuum field of the LSC control system shows a value corresponding to the zero adjustment value currently set (factory setting 0.0010 mbar). The button labeled “adj” can now be used to increment this value up to a maximum of $1.0x10^{-2}$ mbar. As of this value, the output starts again at 0.0010mbar. When the zero calibration value is adapted to the applied zero reference value, the new zero reference value will be saved automatically after 5 seconds if no key is pressed and the calibration will be performed. If from now on the “adj” button is pressed at zero pressure, the system will be calibrated to the set zero calibration value.

If “set” is entered without any other key depression, the zero calibration value will be shown in a purely informative manner for 5 seconds, but no calibration will be performed.

7.4. Rubber valves

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.

7.5. 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 week). Top up oil to the required level via the oil inlet. Due to the continuous operation with gas ballast, oil consumption cannot be avoided. For topping-up see the operating manual of the pump.

Since spring 2004, most of the vacuum pumps delivered by CHRIST have been equipped with an automatic oil recirculation system. This system reuses the oil separated by the exhaust filter to feed the pump. A manual removal is no longer necessary.

The first oil change must be carried out after approx. 100 operating hours. Subsequent oil change intervals depend on the operating conditions. In general, an oil change is necessary after approx. 500 to 1000 operating hours.

The oil change should always be carried out with warm pump.

7.6. 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. The condensate is drained using a waste oil drain screw at the filter.

(Please refer to the separate operating manual!)

7.7. Cleaning

7.7.1. Cleaning the freeze dryer

Use soap 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 freeze dryer or of the drying 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.

7.7.2. Cleaning the accessories

When cleaning accessories, special safety measures must be taken as these are measures to ensure operational safety and reliability.

Chemical reactions as well as stress corrosion (combination of changing pressure and chemical reaction) can affect or destroy the structure of metals and plastic parts. Hardly detectable cracks on the surface will expand and weaken the material without any visible signs. When a visible damage of the surface, a crack, a mark or any other change, also corrosion, is detected, the part (shelf, vessel, drying chamber etc.) must be replaced immediately for safety reasons.

Fans, lid seals, vessels, racks, drying chambers and shelves must be cleaned regularly in order to avoid damage.

Cleaning of accessories should be carried out away from the freeze dryer once a week or preferably after every use.

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

Aluminium accessories are particularly prone to corrosion. A neutral cleaning agent with a pH-value between 6 and 8 should be used for such parts. Alkaline cleaning agents (pH > 8) must be avoided. This increases the service life and considerably reduces the susceptibility to corrosion.

Careful maintenance by the user increases the service life and prevents premature failure of accessories. Damage caused by insufficient care does not constitute a warranty claim.

7.7.3. Maintenance of the aeration valve and the drain valve

Special attention must be paid to the aeration valve and the drain valve. If residues from previous drying processes deposit on them, the freeze dryer may malfunction. Therefore, take care that no product or other residues will get into the pipe to the aeration valve and to the drain valve.

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

All usual disinfectants like, for example, INCIDUR, Melisiptol, Sagrotan, Buraton or Terralin (available through your local laboratory supplier) can be used.

ATTENTION! Check compatibility with the lid and the drying chamber. See also the enclosure “Chemical behavior of PLEXIGLAS® (acrylic glass)”.

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.**

7.7.5. 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.

Furthermore, a ground conductor check must be carried out regularly.

8. Options

8.1. Process control system LPC-32, software

8.2. Process control system LPC-32, software with PC hardware

8.3. Programmer module PGM 30

8.4. Extension unit HED-16 LSC

8.5. LyoLog LL-1 (documentation software)

8.6. Pressure increase test

8.7. Lyocontrol

8.8. Electrical lifting device

Only for ALPHA 1-4/2-4 LSC GAMMA 1-16 LSC / 2-16 LSC and DELTA 1-24 LSC / 2-24 LSC ALPHA 1-4/2-4 LDplus BETA 1-8/2-8LDplus

8.9. Solvent-resistant version

8.10. IQ / OQ

8.11. Cooling bath CB 18-40

8.12. Cooling bath

Subject to technical changes.

9. Enclosures

Declaration of conformity **ALPHA 1-4 / ALPHA 2-4 LSC**

Operating manual of the vacuum pump (only in case of delivery)

Operating manual of the exhaust filter (only in case of delivery)

Brochure "Freeze Dryer for advanced process" (LSC Control System

Accessories catalogue "The Accessories for Freeze Drying"

Chemical Behaviour ®Plexiglas
(material of the drying chamber and the lid)

Disinfection spray INCIDUR

(Material of all parts getting in contact with the different media)

Return declaration

Declaration of contamination

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Chemical Behaviour

PLEXIGLAS® GS
PLEXIGLAS® XT

The stated behaviour was established for the grades PLEXIGLAS GS 215, 218, 221, 222, 224, 231, 233, 240, 245 and 2458 as well as for PLEXIGLAS XT. The extruded materials are, however, attacked more easily by solvents.

For greater chemical resistance, grade PLEXIGLAS GS 209 is recommended.

The data given refer to a test temperature of 23 °C and presuppose stress-free installation.

The behaviour of the material in practice depends largely on the temperature in use. In case of doubt, we advise you to consult us as to the chemical resistance for particular applications.

The results obtained for all products, especially the branded ones, refer to the production batch tested in each case.

Paints, etc.
o Acrylic paints and lacquers
+ Non-aromatic benzines
- Nitrocellulose lacquers
+ Oil paint, pure
- Thinners in general

Antistatics
+ HB 155
+ Antistatic fluid and cleaning agent

Technical baths
+ Electroplating baths
+ Photochemical baths

Building materials and protectives
- Bitumen emulsion
+ Cement
+ Gypsum
o Hot bitumen
+ Mortar
+ Red lead

Chemicals, solvents, etc.
a) General
- Acetic acid, concentrated
o Acetic acid, up to 25%
- Acetone

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+ Alum
+ Aluminium chloride
+ Aluminium oxalate
+ Aluminium sulphate
- Ammonia water
+ Ammonium sulphate
- Amyl acetate
- Aniline
+ Arsenic
+ Arsenic acid
+ Battery acid
- Benzaldehyde
- Benzene
+ Benzine, pure
- Bromine
- 1-Butanol
- Butyl lactate
+ Butyric acid, up to 5%
+ Calcium chloride
+ Calcium hypochlorite
- Carbon disulphide
- Carbon tetrachloride
- Chlorinated hydrocarbons
- Chlorine, liquid
o Chlorine water
- Chloroethyl ether
- Chlorophenol
o Chromic acid
+ Citric acid, up to 20%
+ Copper sulphate
- Cresol
+ Cyclohexane
- Diacetone alcohol
o Diamyl phthalate
- Dibutyl phthalate
+ Diethylene glycol
- Dioxane
- Ether
- Ethyl acetate
- Ethanol, concentrated
o Ethanol, up to 30%
- Ethyl bromide
- Ethyl butyrate
- Ethylene bromide
+ Ferric chloride
+ Ferrous chloride
+ Ferrous sulphate
+ Formic acid, up to 2%
o Formic acid, up to 40%
+ Glycerol
+ Glycol
+ Heptane
+ Hexane
+ Hydrochloric acid
+ Hydrofluoric acid, up to 20%
+ Hydrogen peroxide, up to 30%
+ Iodine, metallic

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+ Lactic acid, up to 20%
+ Magnesium chloride
+ Magnesium sulphate
+ Manganese sulphate
+ Mercury
- Methanol, concentrated
o Methanol, up to 30%
- Methyl ethyl ketone
- Methylated spirits
+ Milk of lime
+ Monobromonaphthalene
+ Nickel sulphate
+ Nitric acid, up to 40%
- Nitric acid, over 40%
+ Oxalic acid
- Perchloroethylene
+ Petroleum
+ Petroleum ether
- Phenols
+ Phosphoric acid, up to 50%
- Phosphorus trichloride
- Phosphorus, white
+ Picric acid, 1% in water
+ Potassium bichromate
+ Potassium carbonate
+ Potassium chloride
+ Potassium cyanide
+ Potassium hydroxide solution
+ Potassium nitrate
+ Potassium permanganate
o 2-Propanol
+ Propylene
- Pyridine
- Silicon tetrachioride
+ Silver nitrate
+ Soap solution
+ Soda
+ Sodium bisulphite
+ Sodium carbonate
+ Sodium chlorate
+ Sodium chloride
+ Sodium hydroxide solution, 30%
+ Sodium hypochlorite
+ Sodium sulphate
+ Sodium sulphide
+ Stannous chloride
+ Stearic acid
+ Sulphur
- Sulphur dioxide, liquid
+ Sulphuric acid, up to 30%
o Sulphurous acid, conc.
+ Sulphurous acid, up to 5%
+ Sulphuryl chloride
+ Tartaric acid, up to 50%
- Thionyl chloride
- Toluene
+ Triethylamine

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- Trichloroacetic acid
+ Turpentine
+ Turpentine substitute
+ Urea, up to 20%
- Xylene
+ Zinc sulphate, aqueous
+ Zinc sulphate, solid

b) Branded products
+ CLOPHEN® T 55, A 60
o DEKALIN®
o FRIGEN® A 12 (CF_2Cl_2)
- GLYBAL® A
+ PALATINOL® K
o PALATINOL® 0, BB new
+ SANGAJOL®
+ TERAPIN®
- TETRALIN®

Disinfectants
a) General
- Carbolic acid
+ Chlor. lime paste
+ Hydrogen peroxide, up to 40%
o Hydrogen peroxide, over 40%
- Iodine tincture, 5%
+ Lugol solution
- Methylated spirits
+ Sublimate

b) Branded products
o ÄTHROL® up to 5%
- BAKTOLAN®, conc.
+ BAKTOLAN®, up to 5%
+ CHINOSOL®, up to 1%
+ CHLORAMIN®, solution
- CHLORAMIN®, suspension
+ ELMOCID GAMMA®, up to 2%
- LYSOFORM®
+ MEFAROL®, up to 1%
+ MERCKOJOD®, up to 1%
+ MERFEN®
+ PERHYDROL®
+ PERODIN®
+ SAGROTAN®, up to 2%
o SAGROTAN®, up to 5%
o VALVANOL®, up to 2%
+ ZEPHIROL®, up to 5%

Fertilisers
+ NITROPHOSKA®, various grades

Fats, oils, waxes
+ Animal
+ Mineral
o Silicone oil
+ Vegetable

Our technical advice on the uses of our materials is given without obligation. The buyer is responsible for the application and processing of our products and is also liable for observing any third-party rights. Technical data concerning our products are typical values.

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Gases and vapours
+ Ammonia
o Bromine vapours, dry
+ Carbon dioxide
+ Carbon monoxide
+ Citygas
o Chlorine vapours, dry
+ Exhaust gases containing HCl
+ Exhaust gases containing HF
+ Exhaust gases containing H_2SO_4
+ Hydrogen sulphide
+ Methane
+ Nitrogen dioxide
+ Nitrogen monoxide
+ Oxygen
+ Ozone
+ Sulphur dioxide, dry

Beverages, etc.
+ Beer, wine
+ Camomile extract
+ Chocolate
+ Fruit juice, milk, coffee
o Spirits, up to 30%
+ Vinegar
+ Water, mineral water

Adhesives and sealants
- Acrylate sealing compound
o All-purpose adhesive
+ Insulating tape
o PATTEX® special-purpose glue
+ PERBUNAN®
o PLEXISOL® adhesive
o PLEXIT®
+ PLEXTOL® adhesive
- Polyurethane sealing compound
+ Sealing strips (EGO-FERM®, TEROSTAT® 81/86)
o Silicone
- Thiokol rubber (one-and-two component)

Cosmetics, etc.
- Camphor
+ DIPLONA® hair oil
+ Face tonic
+ Glycerine
+ Hair setting lotion (PRIMAWELL®)
- Nail varnishes
- Nail varnish removers
+ Ointments
+ Peat water
+ POLYCOLOR®
+ Seawater
+ Soaps
o Sprays

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Plastics
+ Foam plastics
- Foam plastics, plasticised
+ Polyamide
+ Polyethylene
+ PVC
- PVC, plasticised
+ Rubber
- Rubber, plasticised

Foods and spices
+ Aniseed, bay leaf, nutmeg
- Cloves
+ Common salt
+ Honey, pure
+ Ice cream
+ Meat, fish
+ Pepper, cinnamon, onions
+ Pickles

Cleaning agents
a) General
Acids, see under chemicals
- Alcohol, concentrated
o Alcohol, up to 30%
Alkalis, see under chemicals
+ Ammonia solution
- Benzine, mixture, containing aromatics
+ Benzine, non-aromatic
+ Bleach
- Carbon tetrachloride
- Methylated spirits
- Perchloroethylene
+ Petroleum
+ Petroleum ether
+ Soap solution
+ Soda water
- Stain remover
- Trichloroethylene
+ Turpentine
+ Turpentine substitute

b) Branded products
+ AJAX®
+ ANTISTATISCHER KUNSTSTOFFREINIGER UND PFLEGER
+ BFK® cleanser
o BOLIMENT®
+ BÖTTCHERIN®
+ BURMAT®
+ BURNUS®
+ CILLIT-GRÜN®
+ DOR®
+ DOSYL®
+ DOSYLAN®
+ FAKO® Polish
+ FAKO® Polishing Paste
+ FEWA®

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+ FRAPPIN®
+ FÜLLBOX®
+ LAVAPLEX®
+ NULL-NULL®
+ PERSIL®
+ PLEXIKLAR®
+ PRIL®
+ REI®
+ SEIFIX®
- SIDOLIN®
- SPECTROL®
+ SPÜLI®
+ WC-00®

c) Cleaning agents for pipes and tanks
+ CALGONIT® D, DA, S
+ NEOMOSCAN® M, M powder
+ Niroklar GR liquid
+ Niroklar GR powder
+ P3
o P3 basic cleaner
+ P 3-dix

Pesticides
- Sprays (applied directly)
o Sprays (applied in the air)
o Pesticides in aqueous solutions
+ NEXION® stable spray
+ RABOND® stable spray

Protective coatings (strippable)
+ DIEGEL® liquid film 23922
+ KOPPERSCHMIDT® covering paste
o SPRAYLAT

Other substances
+ Urine
- Fuel for petrol engines
o Fuel for diesel engines

The symbols signify:

+ = resistant
o = conditionally resistant
– = not resistant

Note:
The commercial products mentioned in columns b), and especially those marked ®, have been tested on our products just once.
Different results may be obtained if manufacturers change their formulations.

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® = registered trademark

PLEXIGLAS = registered trademark of Röhm GmbH, Darmstadt

Important notice
This is an international English-language information prepared for several markets.
It is essential that the selection of particular materials and their methods of use conform with the requirements of national and local Building Regulations.
The availability of any particular product should be checked with your supplier.

Material Safety Data Sheet according to 91/155/EEC - ISO 11014-1

Incidur

1. Identification of the product and of the company

Identification of the product:

Incidur

Surface disinfectant for medical inventary and other surfaces
Medical Devices class IIa.

Company/undertaking identification:
Ecolab Deutschland GmbH, Postfach 130406,
40554 Düsseldorf, Tel.: 0211/9893-0

The Henkel information service also provides an around-the-clock telephone service on telephone No. ++49-(0)211/797-3350 for exceptional cases.

2. Composition/information on ingredients

Declaration according recommendation 89/542/EEC:
5 - 15 %: anionic surfactants,
below 5 %: nonionic surfactants,

Further ingredients: Antimicrobical agents, cleaning booster, complexing agent, dyestuff, fragrances.

Declaration of ingredients:

8,8 % Glyoxal
Symbol: Xn
R-phrase: 20-36/38-43-68
EINECS: 203-474-9

4,5 % Glutaraldehyde
Symbol: T,N
R-phrase: 23/25-34-42/43-50
EINECS: 203-856-5

1 - 5 % fatty alcohol ethoxylate
Symbol: Xn
R-phrase: 22-41
EINECS: Polymer

5 -15 % Alkylbenzenesulfonate
Symbol: Xi
R-phrase: 38-41
EINECS: 270-115-0

3. Hazards identification of the product

Xn Harmful
R 37/38: Irritating to respiratory system and skin
R 41: Risk of serious damage to eyes
R 42/43: May cause sensitization by inhalation and skin contact R 68/20/22: Harmful: possible risk of irreversible effects through inhalation and if swallowed.

4. First aid measures

after inhalation:
Fresh air, consult doctor if complaint persist.

after skin contact:
Rinse with running water and soap. Skin care. Remove contaminated clothes.

after eye contact:
Immediately flush eyes with copious amounts of running water (for 10 minutes), see an oculist.

after ingestion:
Rinse out mouth, drink 1-2 glasses of water, seek medical advice.

5. Fire-fighting measures

Suitable extinguishing media:
suitable for all regular extinguishing materials

Extinguishing media which must not be used for safety reasons:
none known

Special exposure hazards arising from the product itself, from combustion products or from resulting gases:
none known

Special protective equipment for firefighters:
Wear self-contained breathing apparatus.

6. Accidental release measures

Personal precautions:
Avoid contact with skin and eyes.
Ensure adequate ventilation.

Environmental precautions:
Do not allow large amounts to be released into the sewer system.

Methods of cleaning up/of removing:
Remove mechanically wash away residue with plenty of water;

Other indications:
Dilute small quantities with large amount of water and rinse.

7. Handling and storage

Handling:
no special measures required

Storage:
Store only in the original container.
Do not store at temperatures above 25°C;

Storage Class: VCI-storage class: 10 (BRD)

8. Exposure controls / personal protection

Information on the system design:
No special measures required

Components with specific control parameters:
The product contains glutaraldehyde, CAS-No.: 111-30-8. The relevant German exposure-limit for working place (MAK-value) according to 'TRGS 900' for this substance is 0,1 ppm = 0,4 mg/m3. (STEL-category =1=)
May be harmful if maximum allowable concentration (MAK) is exceeded.

Personal protection:
wash off any dirt that gets onto the skin with lots of soap and water, skin care;
The German accident prevention regulations (UVV) for health service and welfare (BGW BGV C8 and GUV 8.1 the BUK) specify in their Art. 7 the protective clothing to be worn for cleaning and disinfection measures.

Respiratory protection: when processing large amounts

Hand protection: Wear Category III (EN 374) chemical protective gloves made of butyl rubber or nitrile rubber. Please observe the glove manufacturer's instructions on permeability and ruptur times as well as the specific workplace conditions.

Eye protection: goggles which can be tightly sealed

9. Physical and chemical properties

Physical state:	liquid
Colour:	green
Odour:	aldehyde-like
pH:	(undiluted) (20°C) ca. 4,5
Cloudpoint:	< 0 °C
Clarification point:	> 0 °C
Flash point:	aqueous preparation
Relative density:	(20°C) 1,09 g/cm3
Solubility:	(20°C) soluble in water
Viscosity:	(20°C) ca. 15 mPa.s Höppler

10. Stability and reactivity

Conditions to avoid:
No decomposition if used according to specifications

Materials to avoid:
none known if used for its intended purpose

Hazardous decomposition products:
none if used for intended purpose none known

11. Toxicological information

Possible risks of irreversible effects.

Inhalation:
Harmful by inhalation
Irritating to respiratory system

Ingestion:
Harmful if swallowed

Skin contact:
The product is irritant to skin and mucous membranes.

May cause sensitization by inhalation. May cause sensitization by skin contact

Eye contact:
Risk of serious damage to eyes

12. Ecological information

Persistence and degradability:
This product contains surfactants which are at least 90 % biodegradable by reference to the German regulation June 4, 1986.

13. Disposal considerations

Use rest of contents as far as possible according to instructions.
The valid EWC waste code numbers are source-related.
The manufacturer is therefore unable to specify EWC waste codes for the articles or products used in the various sectors.
In case of doubt we will be happy to advise you.
Can be added to materials collection after completely emptying.

14. Transport information

Not a hazardous material according to RID/ADR, GGVS/GGVE, ADNR, IMDG, ICAO-TI/IATA-DGR.

15. Regulatory information

Classification and labelling according to GefStoffV:

Symbols of danger:
Xn Harmful

Ingredients:
Glutaral (Glutaraldehyd), Glyoxal

R-phrases:
R 37/38: Irritating to respiratory system and skin
R 41: Risk of serious damage to eyes
R 42/43: May cause sensitization by inhalation and skin contact
R 68/20/22: Harmful, possible risk of irreversible effects through inhalation and if swallowed.

S-phrases:
S 2: Keep out of reach of children
S 23: Do not breathe spray
S 26: In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
S 36/37/39: Wear suitable protective clothing, gloves and eye/face protection
S 45: In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible)

National prescriptions:

WGK = 2 water-endangering product (manufacturer classification in conformity with calculation method of the German VwVwS of May 17, 1999).

16. Other information

This information is based on our current level of knowledge and relates to the product in the state in which it is delivered. It is intended to describe our products from the point of view of safety requirements and is not intended to guarantee any particular properties.

Full text of the R-phrases indicated by codes in this safety data sheet. The product code/identification/designation is indicated in Section 15.
R 20: Harmful by inhalation.
R 22: Harmful if swallowed
R 23/25: Toxic by inhalation and if swallowed.
R 34: Causes burns
R 36/38: Irritating to eyes and skin.
R 38: Irritating to skin
R 41: Risk of serious damage to eyes
R 42/43: May cause sensitization by inhalation and skin contact.
R 43: May cause sensitization by skin contact.
R 50/53: Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
R 68: Possible risks of irreversible effects

Declaration of Decontamination / Return Declaration

Following declarations serve for keeping safety and health of our employees. Fill in the forms and attach them when returning freeze dryers, centrifuges, spare parts and accessories. Please understand that we cannot carry out any work before we have the declarations. (We recommend to make several copies of this page.)

!!!! Attention – This form must be glued on outside of the packing !!!!

Return declaration

	YES	NO
Decontamination declaration inside :		
Unit / component contaminated :		
Unit / component unused (new) :		

!!!! Attention – This form must be glued on outside of the packing !!!!

Declaration of Contamination of Freeze Dryers, Vacuum-Concentrators, Centrifuges, Accessories and Vacuum Pumps

This declaration may only be filled in and signed by authorised staff.

Repair Order dtd. : _____

Order No. : _____

Type of unit : _____ Serial No. : _____

Type of unit : _____ Serial No. : _____

Type of unit : _____ Serial No. : _____

Type of unit : _____ Serial No. : _____

Accessories : _____

_____

Is the equipment free from harmful substances ? YES O NO O

If not, which substances have come into contact with the equipment?

Name of the substances : _____

_____

Remarks (e.g. to be touched with gloves only) : _____

_____

General characteristics of the substances :

Corrosive	O	Explosive	O
Biologically hazardous	O	Radioactive	O
Toxic	O		

In combination with which substances may hazardous mixtures develop?

Name of the substances : _____

_____

Has the equipment been cleaned before shipment? YES O NO O

Is the equipment decontaminated and not harmful to health? YES O NO O

Prior to repair, radioactively contaminated components must be decontaminated according to the valid regulations for radiation protection.

Legally Binding Declaration

I / we hereby declare that the information on this declaration are correct and complete.

Company / Institute : _____

Street : _____

Postcode, City : _____

Tel. : _____ FAX : _____

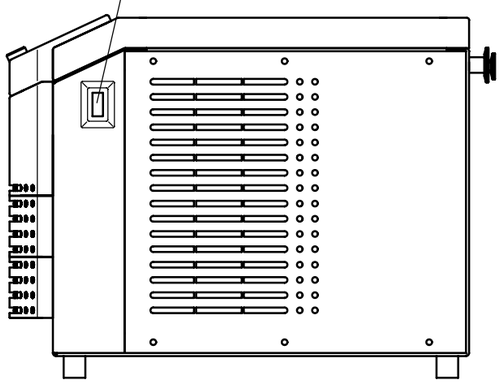
Name : _____

Date : _____ Stamp :

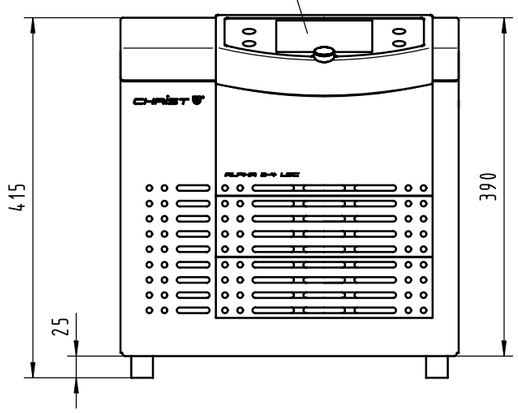
Signature : _____ _____

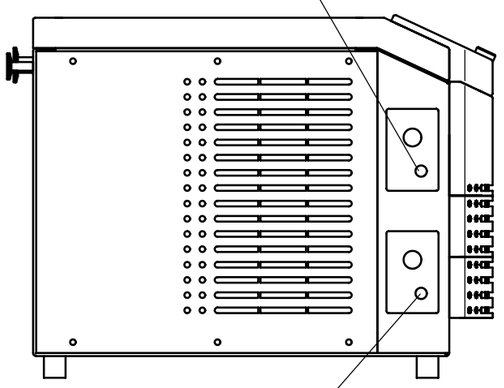
10. Special functions

Netzschalter
Mains switch
Commutateur d'alimentation



Anlagensteuerung LSC
Control system LSC
Système de contrôle LSC





Belüftungsventil
Aeration valve
Soupape de ventilation

Ablassventil
Drain valve
Vanne de vidange

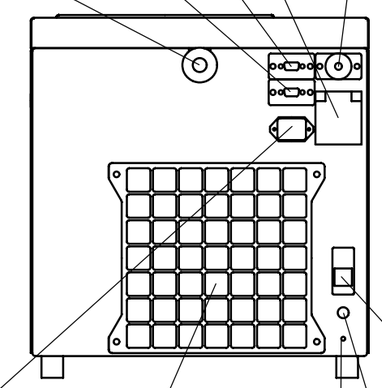
serielle Schnittstelle Vakuummesssonde
Serial interface of vacuum measuring sensor
Interface sérielle du capteur de vide

serielle Schnittstelle
serial interface
Interface sérielle

Anschluss Spannungsversorgung Vakuumpumpe
Vacuum pump power supply connection
Connexion de l'alimentation électrique de la pompe à vide

Anschluss Vakuummesssonde
Vacuum measuring sensor connection
Connexion du capteur de vide

Vakuumanschluss
Vacuum connection
Connexion de vide



Wärmetauscher der Kälteanlage
Heat exchanger of refrigeration unit
Echangeur thermique du groupe frigorifique

Netzabsicherung
Mains fuse
Fusible

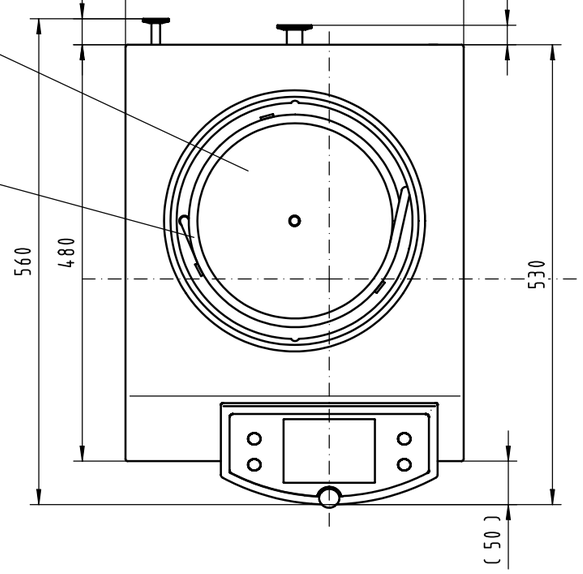
Netzkabel
Mains cable
Cordon d'alimentation

Anschluss Spannungsversorgung Drucksteuer- und Absperrventil
Power supply connection of pressure control and stop valve
Connexion de l'alimentation électrique de la vanne régulatrice de pression et de la vanne d'arrêt

Potentialausgleichsschraube für Schutzleiterprüfung und zentraler Erdungsanschluss
Equipotential bonding screw for protective ground wire check and central ground connection
Vis d'équilibrage de potentiel pour le contrôle du conducteur de protection et pour la connexion centrale de terre.

Eiskondensatorkammer
Ice condenser chamber
Piège

Eiskondensator
Ice condenser
Condenseur



Schutzvermerk nach
DIN 34 beachten!

Verwendungsbereich					Oberflächen DIN ISO 1302	Allgemeintoleranzen DIN ISO 2768 -mk-	Maßstab 1:5 / Gewicht
							Werkstoff, Halbzeug / Rohteil-Nr
					Datum	Name	Benennung: Aufstellplan Layout / Plan d'arrangement ALPHA 1-4 / 2-4 LSC
				Bearb	26.04.2006	R. Gründel	
				Gepr	22.06.2006	M. Umbach	
				Norm			
				MARTIN CHRIST 37520 Osterode			Zeichnungsnummer: 176277 / Blatt 1 / 1 Bl
06.236	Zeichnung aktualisiert	22.06.2006	Gr				
Zust	Änderung	Datum	Name	Urspr			Ers f / Ers d