

Delta 1-4 LSCplus

Part no. 102300, 102302, 102304, 102306, 102308, 102310, 102316, 102318, 102320, 102322

Delta 2-24 LSCplus

Part no. 102301, 102303, 102305, 102307, 102309, 102311, 102317, 102319, 102321, 102323



Operating Manual

Please retain for later use!





In case of inquiries, please state the following numbers:

Order number:

Serial number:

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1 General information

1.1 Importance of the operating manual

A fundamental requirement for the safe and trouble-free operation of the unit is to be familiar with the fundamental safety instructions and all possible hazards.

The operating manual includes important information concerning the safe operation of the freeze-dryer.

This operating manual, and in particular the notes on safety and hazards, must be observed by all persons operating the unit.

In addition, the local rules and regulations for the prevention of accidents must be complied with.

1.2 Intended use

CHRIST freeze-dryers have been solely designed for the freeze-drying of solid or liquid products in ampoules, vials or dishes. They are, therefore, solely intended for these applications.

The freeze-dryer is suitable

- for freeze-drying materials such as bacteria and virus cultures, blood plasma, serum fractions, antibodies, sera, vaccines, and pharmaceutical products
- for freeze-drying plant extracts, e.g. for biochemical tests
- for freeze-drying inorganic materials, e.g. nanoparticles
- for freeze-drying numerous other products (for further information please contact our qualified personnel)

Any other use beyond this area of application is regarded as improper use. Martin Christ Gefriertrocknungsanlagen GmbH cannot be held liable for any damage resulting from such improper use.

The intended use also includes:

- observation of all the notes and instructions included in the operating manual
- compliance with the inspection and maintenance instruction
- prohibition of any type of extensions to, or conversions of the unit.

1 General information



1.3 Warranty and liability

The warranty and liability are subject to our "General Terms and Conditions" that were distributed to the operator upon the conclusion of the contract.

Warranty and liability claims are excluded if they are due to one or several of the following reasons:

- improper use
- non-compliance with the safety instructions and hazard warnings in the operating manual
- improper installation, start-up, operation, and maintenance of the freeze-dryer.

1.4 Copyright

The copyright concerning the operating manual remains with Martin Christ Gefriertrocknungsanlagen GmbH.

The operating manual is solely intended for the operator and their personnel. It includes instructions and information that may not be

- duplicated,
- distributed, or
- communicated in any other way neither in full nor in parts.

Non-compliance may be prosecuted under criminal law.

1.5 Explanation of symbols

In this operating manual, specialist terms that are explained in the glossary (see chapter 12 - "Glossary") are marked by an arrow and printed in italics (e.g. \rightarrow *sublimation*).

1.6 Standards and regulations

EC declaration of conformity in accordance with the EC Machinery Directive (see chapter 11.3 - "EC declaration of conformity in accordance with the EC Machinery Directive")

1.7 Scope of supply

The scope of supply comprises:

- 1 tube of high-vacuum grease
- 1 litre of vacuum pump oil
- 1 set of flange components and several small parts for service and maintenance purposes
- 1 drain hose 0.5 m (silicone 8 x 12 mm)
- 1 operating manual

Accessories and commissioning

According to your order, our order confirmation, and our delivery note.



2.1 Layout of the freeze-dryer

2.1.1 Functional and operating elements

- LSCplus user interface (see chapter 6.5.1 -1 "User interface")
- 2 Vacuum pump

Touchpanel

4

Oil mist separator 3



Fig. 1: Front of the freeze-dryer



Fig. 2: User interface with touchpanel

- 5 Contact bolt
- 6 Pipe connection of the vacuum pump (behind the cover plate)
- 7 Ice condenser
- 8 Vacuum sensor

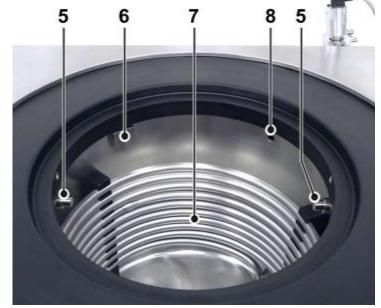


Fig. 3: Ice condenser chamber

Control system on/off 9 (mains power switch) 9 10 Steerable castors

Fig. 4: Right side



7 -	—10—	-1	
		_	
of the freeze	e-dryer		



- 11 Aeration valve
- 12 Media drain valve
- 13 Fixed castors

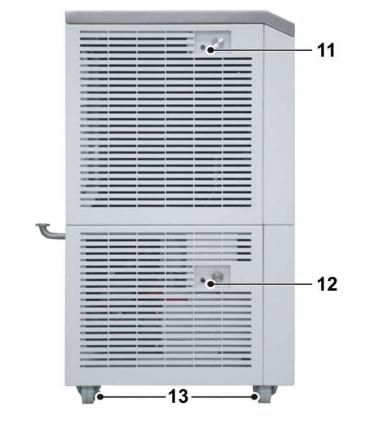


Fig. 5: Left side of the freeze-dryer

- 14 Vacuum sensor
- 15 Mains voltage (Main switch)
- 16 Electrical connection of the vacuum sensor
- 17 Serial interface
- 18 Mains cable
- 19 Heat exchanger of the refrigeration unit (behind the panel)
- 20 Option: Connection for electrical lifting device
- 21 Name plate (see chapter 2.1.2 - "Name plate")
- 22 Vacuum connection

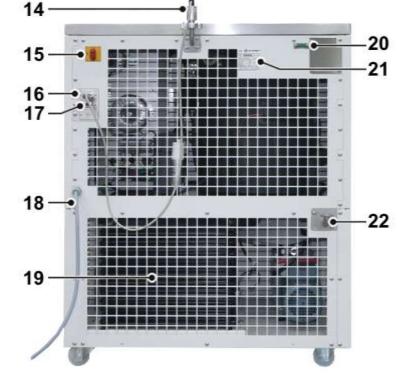


Fig. 6: Rear side of the freeze-dryer



2.1.2 Name plate

- 1 Serial number
- 2 Type
- 3 Refrigerant data of the 1st stage
- 4 Nominal voltage
- 5 Year of manufacture (month/year)
- 6 Part number
- 7 Refrigerant data of the 2nd stage
- 8 Rated current / apparent power

Serial OC	0000	05 / 201
Delta 2-24 LS	SCplus	102301
cool. system	1. stage	2. stage
refrigerant	R404A	R290 / R50
filling	570g	10g / 150g
max. pressure	25 bar	245bar
max. temp.	120°C	120°C

Fig. 7: Example of a name plate (here: Delta 2-24 LSCplus)



2.2 Mode of operation

2.2.1 General information on freeze-drying

What is freeze-drying?

Freeze-drying or lyophilisation is a procedure for the gentle drying of highquality products. The product is dried by \rightarrow *sublimation* without passing through the liquid phase.

What are typical applications for freeze-drying?

As far as their sheer quantity is concerned, foodstuffs are the major application for freeze-drying. One widely known example is the production of granulated instant coffee or the drying of fruit, e.g. for breakfast cereals. Other areas of application are the restoration of water-damaged documents or the drying of archaeological artefacts.

Another important area of application is the drying of biotechnological and pharmaceutical products, e.g. tissues and tissue extracts, bacteria, vaccines, and sera. Products that would not keep well when they are dissolved in water can be preserved by freeze-drying. During this process, the biological properties of these sensitive substances are preserved. The compounds remain unchanged from a qualitative and quantitative point of view. After the addition of water, the products will have the same characteristics as the original products.

How does freeze-drying work?

Freeze-drying is a very gentle procedure for the extraction of water from a product in the frozen state. The drying process takes place through \rightarrow *sublimation*, i.e. the direct transition of a product from the solid phase to the gas phase. This happens under vacuum.

The following section describes the process of sublimation based on the example of water, since most products that are processed by freeze-drying are aqueous solutions. Their behaviour is based on identical fundamental principles.

The vapour pressure curve above ice describes the phase transition as a function of the pressure and temperature. The higher the temperature is, the higher the vapour pressure.

- If the vapour pressure is higher than 6.11 mbar (A), water passes through all three phases: solid, liquid, and gas (see the illustration).
- If the vapour pressure is below 6.11 mbar (B) and energy is added, the ice will be directly converted into water vapour once the sublimation curve is reached. This transition is called "sublimation". If thermal energy is added to pure ice with a temperature of less than -30°C at a pressure of 0.37 mbar, it will be converted into water vapour once it reaches -30°C (see figure).

The vacuum prevents the melting of ice when energy is added. If thermal energy is added to a frozen product under vacuum, thawing of the product will be prevented and the water that is contained within the product will be released in the form of water vapour.



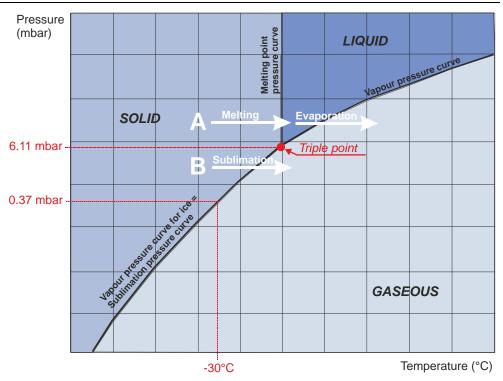


Fig. 8: Vapour pressure curve above ice

From a physical point of view, the freeze-drying process covers three phases (see figure below):

(1) Freezing: The product to be dried is frozen under atmospheric pressure. This can be done either directly in the freeze-dryer or in a separate deep-freeze. The freezing temperature should be approximately 10°C below the solidification point of the product.

(2) Evacuation: When the product is sufficiently frozen, the vacuum pump is activated. The pressure inside the drying chamber will be lowered to the value that corresponds to the freezing temperature in accordance with the vapour pressure curve above ice.

(3) Sublimation: Thermal energy is added to the product, thus starting the sublimation process. Due to the added energy, the water in the product is converted into water vapour. Since the ice condenser is much colder than the product that is to be dried, the vapour pressure in the ice condenser is considerably lower than above the product. As a result, the water vapour that is released by the product streams to the ice condenser, where it condenses on the condenser coils.

Once the free water has been extracted from the product during the main drying phase, the last traces of bound water will also be removed at a final pressure that is as low as possible and at higher temperatures. This takes place by way of \rightarrow *desorption*. This drying phase is also called final drying.



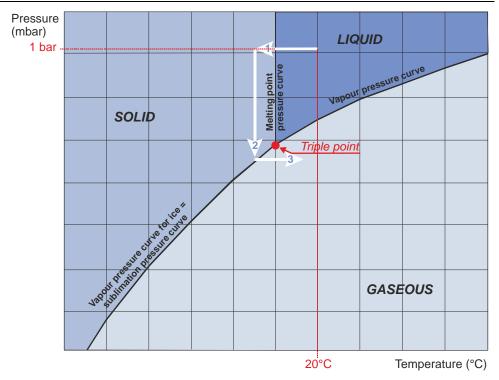


Fig. 9: Freeze-drying phases



Please find further information about basic principles, optimum procedures and applications in the brochure "Smart freeze-drying", which can be downloaded at <u>www.martinchrist.de</u> \rightarrow [Applications] \rightarrow [Lyophilisation].



2.2.2 Freeze-drying process

The main components of a freeze-dryer are:

- vacuum drying chamber with a temperature control system for adding thermal energy
- vacuum pump for generating a vacuum inside the drying chamber
- ice condenser for binding the water vapour that is released by the product.

2.2.2.1 Preparation

The ice condenser chamber must be clean and dry. Any water residues from a preceding drying run must be removed.

The media drain valve and the aeration valve must be closed.

In the case of units that are equipped with a pressure control valve (standard on LSCplus units), the vacuum pump should be warmed up ("warm-up") for at least 15 minutes prior to the start of the main drying phase. Do not subject the vacuum pump to condensable gases until the operating temperature is reached. In this way, the service life of the vacuum pump can be extended.

At the same time, the ice condenser is pre-cooled ("cool-down"). The ice condenser temperature does not have any influence on the product temperature. The sole purpose of the ice condenser is to bind the released water vapour.

2.2.2.2 Freezing

First, the product that is to be dried is frozen. This can be carried out either directly in the freeze-dryer or in a separate deep-freeze. Especially in the case of small filling quantities, we recommend pre-cooling the shelves as well in order to prevent the product from thawing during the evacuation.

Two very different structures of the frozen material can be distinguished:

- · crystalline structures with clearly distinguishable crystals
- amorphous structures with no crystal junctions at all (e.g. glass)

The majority of the freeze-drying products have a crystalline form.

When freezing these kinds of products, one must take into consideration that too deep and too quick freezing leads to smaller ice crystals, which has a negative effect on the duration of the drying process.

For every product to be dried, the solidification point must be determined as a first step. This is the point at which the water that is contained in the product has completely crystallised. In order to ensure an optimum freezedrying process, the product temperature should be approximately 10°C below the solidification point.

A layer thickness of the product of 1-2 cm should not be exceeded, since otherwise the drying duration would be negatively affected. If liquids are to be dried in bottles with a layer thickness of more than 1 cm, we recommend freezing them in a cooling bath with the aid of a shell or spin freezing device (see figure). Due to the centrifugal force, the liquid to be frozen will rise on the inner wall of the bottle and freeze. This procedure reduces the layer thickness and, thereby, the total drying time will be shortened to a considerable extent (see figures on the right side).





Fig. 11: Cooling bath with spin freezing device Fig. 12: Spin freezing



If the product that is to be dried contains solvents or high salt concentrations, it may start to thaw during the drying process, which is indicated by clearly visible foaming. In order to prevent this, the product must be frozen as deeply as possible, e.g. with the aid of liquid nitrogen, prior to putting it into the unit.

2.2.2.3 Main drying

When the product is frozen to a sufficiently deep extent, the main drying phase commences. The vacuum pump is switched on. The pressure inside the drying chamber will be lowered to the value that corresponds to the freezing temperature in accordance with the vapour pressure curve above ice. At the same time, thermal energy will be added to the product. In the case of products in round-bottom flasks, wide-neck bottles, etc., this is realised through the environment that is considerably warmer (direct contact heat), in the case of unheated shelves by way of thermal radiation from the environment, and in the case of temperature-controlled shelves directly via the shelves. As a result, the sublimation process starts.

At the beginning of the drying process, the maximum drying rate will be reached. The more the sublimation area recedes into the product, the further the produced water vapour must pass through the layers that have already been dried.

Under certain conditions, it is possible that the vacuum inside the ice condenser chamber increases during the main drying phase (e.g. from 0.63 mbar to 0.47 mbar) although the valve towards the vacuum pump is closed. From a physical point of view, this is due to the pumping effect of the ice condenser ("cryo-pumping effect").

The required drying time depends strongly on the drying vacuum. At 1.0 mbar, one gram of ice takes up a volume of 1 m^3 of vapour, at 0.1 mbar a volume of 10 m^3 of vapour, and at 0.001 mbar a volume of 100 m^3 . The closer the vacuum is to the solidification point, the smaller is the resulting vapour volume. The drying rate increases and the drying time decreases.

The end of the main drying phase is reached, when the product temperature and the shelf temperature are nearly identical. The temperature difference between the shelf and the product should be approximately 3 K to 5 K.



2.2.2.4 Final drying

Final drying is an option whenever one requires a product with minimal residual moisture. In the physical sense, this process is a desorption process, i.e. the removal of adsorptively bound water. Final drying is performed under the lowest possible final pressure that depends on the ice condenser temperature in accordance with the vapour pressure curve above ice as well as on the final vacuum of the vacuum pump that is used. The process is supported by a higher shelf temperature.

2.2.2.5 End of drying and aeration

The end of the drying process is reached when both the product and shelf temperature are clearly in the positive range (+15 to +20°C) and if their difference is not greater than 5 K.

Another indication of the end of the drying process is the behaviour of the vacuum and of the ice condenser temperature. The ice condenser is no longer subject to load and reaches the final temperature of approximately - 55°C or -85°C. The pressure in the drying chamber decreases in accordance with the ice condenser temperature.

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

Then, the product can be removed from the unit.

2.2.2.6 Defrosting

Defrosting with hot gas

As standard, the freeze-dryer is equipped with a hot-gas defrosting system. In order to defrost the ice condenser, heated refrigerant is fed through the heating coil. In addition, the bottom of the ice condenser chamber is heated by way of a heating collar.

In order to avoid damage, the condensate must be drained off through the media drain valve directly after the completion of the defrosting process. Then, any residual water must be removed from the ice condenser chamber by way of a cloth.



Special equipment: defrosting with hot water



1

Defrosting ring

For hot-water defrosting, the freeze-dryer must be connected to a hot water supply.

The ice condenser is defrosted by feeding hot water into the ice condenser chamber. The hot water is sprayed onto the ice condenser until the maximum filling level is reached in the ice condenser chamber. Then, the feed flow will be stopped automatically and the drain valve will be opened until the liquid level in the ice condenser chamber falls below a certain level. This process will be repeated at certain intervals until the preselected defrosting time has elapsed.

In order to avoid damage, any residual water must be removed from the ice condenser chamber directly after the completion of the defrosting process by way of a cloth.

Fig. 13: Ice condenser chamber with a hot-water defrosting system

3 Safety



3 Safety

3.1 Marking of the unit

The following symbols are used for CHRIST freeze-dryers:

	Dangerous voltage	I.	On (Power)
	Hot surface	0	Off (Power)
	Caution! Risk of bruising		Name plate (see chapter 2.1.2 - "Name plate")
	Attention, consult the operating manual	CE	CE mark in compliance with the directive 2006/42/EC
	Protective earth (ground)	B	Unplug the mains plug
Ţ	Earth (ground)	HIB EH / Mag. Dam 3333 - MM - DO	China RoHS 2 mark (only for China)



Safety indications on the freeze-dryer must be kept readable at all times. If necessary, they must be replaced.



Not all of the symbols/labels are used for this type of freeze-dryer.



3.2 Explanation of the symbols and notes

This operating manual uses the following names and symbols to indicate hazards:



This symbol stands for a **direct** hazard to the life and health of persons.

Non-observance of these symbols causes serious health problems up to life-endangering injuries.



This symbol stands for a **<u>direct</u>** hazard to the life and health of persons due to electrical voltage.

Non-observance of these symbols causes serious health problems up to life-endangering injuries.



This symbol stands for a **potential** hazard to the life and health of persons.

Non-observance of these symbols <u>can</u> cause serious health problems up to life-endangering injuries.



This symbol indicates a potentially hazardous situation

Non-observance of these notes can cause minor injuries or damage to property.



This symbol indicates important information.



3.3 Responsibility of the operator

The operator is obliged to ensure that the persons working with the freezedryer

- are 18 years old or older,
- have been specifically ordered to do so by the operator and that they
 have been duly informed about the specific hazards associated with the
 system, supply media and starting/final products as well as about the
 correct conduct and necessary measures to take in the event of
 accidents or malfunctions,
- are familiar with the fundamental regulations concerning workplace safety and accident prevention,
- have read and understood this operating manual (and in particular the safety sections and warning notes) and confirmed this with their signature.

The areas of responsibility of the personnel concerning the operation, maintenance, and care of the unit must be clearly defined.

The safety-conscious work of the personnel in compliance with the operating manual and the relevant EC and national health and safety regulations as well as with the accident prevention regulations must be checked at regular intervals (e.g. every month).

The operator is responsible for performing a risk assessment in terms of disasters (e. g. fire) in the working area and, if necessary, for taking constructional measures.

The operator is responsible to check the chemical compatibility of all substances to be used inside the freeze-dryer (product to be processed, cleaning media, etc.) with the material of the chamber walls, shelves, pipes, and gaskets. Substances that may damage the material an degrade the mechanical strength must not be used.

The freeze-dryer has to be maintained regularly (see chapter 8 - "Maintenance and service").

Components that are not in a perfect state must be replaced immediately.

3.4 Operating personnel

It must be ensured that persons operating the unit

- are 18 years old or older,
- have been specifically ordered to operate the unit and made aware of dangers originating from the freeze-dryer, supply media, starting and end products by the operator,
- be familiar with the fundamental regulations concerning workplace safety and accident prevention
- · have been trained in terms of the operation of this unit, and
- have read and understood this operating manual (and in particular the safety sections and warning notes) and confirmed this with their signature.



3.5 Informal safety instructions

This operating manual is a part of the product.

- The operating manual must be kept at the location of use of the freezedryer. Ensure that it is accessible at all times.
- The operating manual must be handed over to any subsequent owner or operator of the freeze-dryer.
- Any changes made must be added to the operating manual.
- In addition to the operating manual, the general and local rules and regulations concerning the prevention of accidents and the protection of the environment must also be supplied.
- Safety and danger indications on the freeze-dryer must be kept readable at all times. If necessary, they must be replaced.

3.6 Safety instructions

The following instructions must be observed prior to every drying process:



- Ensure that the freeze-dryer was set up and connected properly (see chapter 5 "Set-up and connection").
- Connections to customer-provided pipes must be force- and torquefree.
- Maintain a safety distance of at least 30 cm (12 inches) around the freeze-dryer.
- Do not store any dangerous goods in the safety area of the freezedryer.
- Do not stay in the safety area longer than what is absolutely necessary for the operation of the freeze-dryer.
- Only use accessories that have been approved by the manufacturer (except for commercial vessels made of glass or synthetic materials).
 We explicitly warn against the use of equipment of poor quality.
 Breaking glass or bursting vessels can cause dangerous situations.
- Observe the instructions on the installation of accessories (see the separate document).

3 Safety



3.6.1 Electrical safety

CHRIST freeze-dryers are units of safety class I. Please comply with the following points in order to preserve the safety features:



- Ensure that the local mains voltage matches the nominal voltage that is stated on the name plate.
- Do not place any dangerous material, e.g. glass vessels containing liquid substances, within the safety area of 30 cm around the freezedryer. Spilled liquids may get into the freeze-dryer and damage the electrical or mechanical components.
- Work on the power supply system must only be performed by certified electricians.
- Inspect the electrical equipment of the unit regularly. Defects such as loose or burnt cables must be eliminated immediately.

3.6.2 Mechanical safety

In order to ensure the safe operation of the freeze-dryer, please comply with the following points:



- Do not use the freeze-dryer if it was installed incorrectly.
- Do not use the freeze-dryer without panels.
- Do not use the freeze-dryer with accessories that shows signs of damage.
- Only use the freeze-dryer with accessories that have been approved by the manufacturer. In case of doubt, contact the manufacturer (see chapter 7.3 "Service contact").
- Do not hit or move the freeze-dryer during its operation.
- Do not lean against or rest on the freeze-dryer during its operation.
- Check the freeze-dryer and the accessories before every start-up for any visible signs of damage.
- Do not dry any substances that could damage the material of the ice condenser chamber, drying chamber, lid, or accessories in any way, e.g. highly corrosive substances such as hydrogen chloride (HCI).
- Stop the freeze-dryer immediately in the event of a malfunction. Eliminate the malfunction (see chapter 7 - "Malfunctions and error correction") or contact the after-sales service of Firma Martin Christ Gefriertrocknungsanlagen GmbH (see chapter 7.3 - "Service contact").
- Ensure that all repairs are performed only by authorised and specialised personnel.



3.6.3 Fire prevention

Fuses protect certain electrical circuits within the freeze-dryer against overcurrent conditions.



- Always use fuses of the same type and rating.
- Do not dry explosive or inflammable substances.
- Do not use the freeze-dryer within hazardous locations where there is a risk of explosion.

3.6.4 Thermal safety

During the operation of the freeze-dryer, the ice condenser can reach surface temperatures of -85° C depending on the power.



Wear suitable protective gloves when installing or removing accessories. Do not reach into the ice condenser chamber without this protection – your limbs may freeze onto the chamber walls!

3.6.5 Chemical and biological safety

If infectious, toxic, pathogenic, or radioactive substances are intended to be dried, it is in the responsibility of the user to ensure that all necessary safety regulations, guidelines, precautions, and practices are adhered to accordingly.

- Infectious, toxic, pathogenic, and radioactive substances must be dried in suitable accessories. Take suitable precautions for your own safety!
- Do not dry products with a high solvent concentration or acidic products without special protective measures or device-based precautions (e.g. an additional cooling trap to protect the vacuum pump). It is absolutely necessary to consult the manufacturer in these cases (see chapter 7.3 "Service contact").
- Special caution is necessary when handling azides, as a dangerous explosive develops in combination with copper or nonferrous metals! It is absolutely necessary to contact the manufacturer (see chapter 7.3 "Service contact").



DANGER

- Keep informed about local measures to avoid harmful emissions (depending on the substances to be dried).
- As personal protective equipment, safety gloves are required for the use of the freeze-dryer.
 The materials to be dried may, however, require additional special safety measures (e.g. drying of infectious, toxic, radioactive, or

pathogenic substances).

3 Safety



3.7 Safety devices

3.7.1 System check

An internal system check system monitors the data transfer and sensor signals with regard to plausibility. Errors are detected by continuous self-monitoring of the system. Error messages are displayed in the main window under "Process & equipment messages" (chapter 6.5.1.1 - "Main window "Manual"", chapter 7.2 - "Process and error messages").

3.8 Procedures in the event of hazards and accidents

Hazardous electrical incident:

• Set the main switch and the mains power switch of the control system to the "OFF" or "0" position in order to interrupt the power supply completely.

Fire:

- A fire in the electrical control system must be extinguished with a CO₂ fire extinguisher!
- Burning oil must be extinguished with a CO₂ fire extinguisher or powder fire extinguisher!

Electric shock:

 While ensuring your own safety, interrupt the circuit as quickly as possible (main switch and control switch). Keep the affected persons warm and calm. Get medical attention immediately! Check consciousness and breathing continuously. In the case of unconsciousness of lack of normal breathing, perform cardiopulmonary resuscitation (CPR).

Burns:

- Cool small-area burns (e.g. finger) immediately with cold water for approximately 2 minutes.
- Do not cool if larger areas of the body surface are burnt since there is a risk of hypothermia.
- Cover the burns loosely and in a sterile manner (e.g. with sterile dressing).
- Keep the affected persons warm and calm.

IF IN DOUBT, CALL THE EMERGENCY PHYSICIAN (AMBULANCE)!



3.9 Remaining hazards

All CHRIST freeze-dryers were built state-of-the-art and according to the accepted safety rules. Danger to life and limb of the operator, or of third parties, or impairments of the units or other material assets, however, cannot be completely excluded when the units are being used.

Use the freeze-dryer

- only for the purpose that it was originally intended for (see chapter 1.2 -"Intended use") and
- only if it is in a perfect running state.
- Immediately eliminate any problems that can affect safety.

4 Storage and transport



4 Storage and transport

4.1 Dimensions and weight

Values for the freeze-dryer without a vacuum pump:

	Delta 1-24 LSCplus	Delta 2-24 LSCplus
Height:	1.090 mm	1.090 mm
Width:	860 mm	860 mm
Depth:	610 mm + 80 mm vacuum connection	610 mm + 80 mm vacuum connection
Weight:	approx. 215 kg	approx. 250 kg

4.2 Storage conditions

In order to ensure the protection against mechanical and climatic influences, the guidelines of the German Federal Association for Wooden Packages, Pallets, and Export Packaging (Bundesverband Holzpackmittel, Paletten, Exportverpackung e.V.), the so-called HPE packaging guidelines, must be applied when packing and storing the freeze-dryer.

The storage must be:

- dust-free
- dry
- · free from excessive temperature fluctuations
- free from a mechanical load.

4.3 Notes on transport

CAUTION

CAUTION

- Use suitable packaging for the transport, and if at all possible, the original packaging.
- Install all transport safety devices (see chapter 4.5 "Transport safety devices").

The freeze-dryer **Delta 1-24 LSCplus** weighs approx. 215 kg! The freeze-dryer **Delta 2-24 LSCplus** weighs approx. 250 kg!

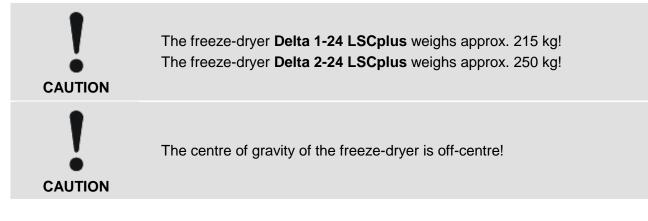
The centre of gravity of the freeze-dryer is off-centre!



4.4 Packaging

The freeze-dryer is packaged in a wooden crate.

- After opening the packaging, take out the accessories.
- Remove the packaging material.
- Remove the side walls of the crate.
- Lift the freeze-dryer upwards with a lifting device (see chapter 4.6 "Onsite transport").



• Retain the packaging for any possible future transport of the freezedryer.

4.5 Transport safety devices

The following transport safety devices must be removed prior to start-up:

• Install the vacuum sensor (see chapter 5.5 - "Vacuum sensor").



Prior to any transport, the transport safety devices must be reinstalled.

4 Storage and transport



4.6 On-site transport

The freeze-dryer comes with castors. It can be pushed by a suitable number of persons over short distances. Over longer distances, it should be transported by way of suitable lifting equipment (e.g. forklift truck).

- Lift the freeze-dryer upwards with a lifting device. Always reach under the freeze-dryer from the front side (A).
- 1 Steerable castors
- 2 Supports for transport
- 3 Supporting frame
- 4 Fixed castors
- A Front side

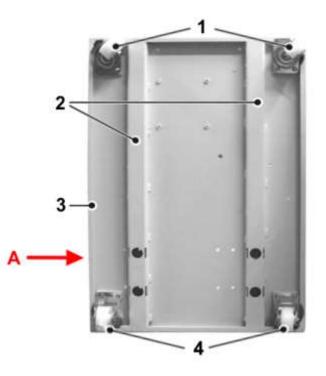
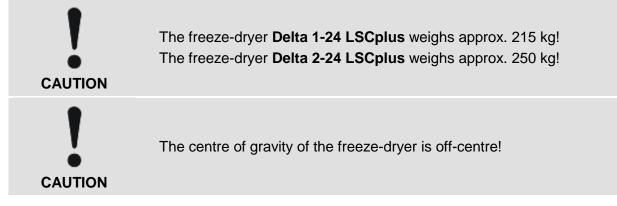


Fig. 14: Bottom of the freeze-dryer





5 Set-up and connection

5.1 Installation site

Operate the freeze-dryer only in closed and dry rooms.



Refrigeration problems of the freeze-dryer are often caused by insufficient conditions at the location of use. This is why compliance with the following conditions is absolutely mandatory!

- The table must be stable and have a solid, even tabletop.
- Ensure sufficient ventilation. Do not place any paper, cloth, or similar material behind or under the unit, since otherwise the air circulation will be impaired.
- Keep a safety distance of at least 30 cm around the freeze-dryer so that the vents in the unit remain fully effective.
- The ambient temperature must be in the range of +10°C to +25°C. A potential night-time setback of the air conditioning system must be taken into consideration.
- Prevent the room temperature from rising, for example due to closed doors at night.
- Do not subject the freeze-dryer to thermal stress, e.g. by positioning it near heat generators.
- Prevent thermal overload, e.g. caused by other equipment in the direct vicinity of the freeze-dryer.
- Do not set up the vacuum pump directly next to the heat exchanger (condenser).
- In the case of water-cooled systems, ensure that the water circuit provides a sufficient amount of cooling water.
- Avoid direct sunlight (UV radiation).

5 Set-up and connection



5.2 Power supply

5.2.1 Connection



The operating voltage on the name plate must correspond to the local supply voltage!



Work on the power supply system must only be performed by certified electricians.

CHRIST freeze-dryers are units of safety class I. The freeze-dryers of the type **Delta 1-24 LSCplus** and **Delta 2-24 LSCplus** have a five-wire power cord with a 5-pin Cekon-plug (see chapter 10 - "Technical data").



Prior to connecting the unit to the power supply system, check the rotary field at the electrical outlet with a suitable measuring instrument:

The rotary field must be clockwise!

5.2.2 Customer-provided fuses

Typically, the freeze-dryer must be protected with 16 Amp G fuses that are to be provided by the customer.

5.3 Aeration valve

The aeration valve is located on top of the left side of the unit (see chapter 2.1.1 - "Functional and operating elements").

After the end of a freeze-drying process, the unit will be aerated via the aeration valve.



The ice condenser chamber can be flooded with nitrogen via the hose nozzle of the aeration valve.



For the aeration with gases under pressure, the client must install a safety device for pressure relief.



5.4 Media drain valve

The media drain valve is located at the bottom of the left side of the unit (see chapter 2.1.1 - "Functional and operating elements").

It is used to drain off the condensate and the defrosting water.

- Connect the drain hose (included in the scope of supply) to the hose connector.
- Place a collecting vessel under the unit.

The hose must be laid with a continuous slope and the end of the hose must always be above the liquid level in the collecting vessel. This prevents water and dirt residues from being sucked into the ice condenser chamber if there is negative pressure when the media drain valve is opened.

5.5 Vacuum sensor



Please refer to the separate operating manual of the vacuum sensor!

In order to protect the vacuum sensor against transport damage, it comes supplied in its original packaging. Prior to commissioning the freeze-dryer, the sensor must be installed.

- 1 Vacuum sensor
- 2 Connection socket



Fig. 15: Position of the vacuum sensor and the connection socket

- Switch the unit off by actuating the mains power switch.
- Take the vacuum sensor out of its original packaging and fasten it to the connector with a clamping ring (DIN16KF) and a centring ring (included in the scope of supply).
- Plug the connector to the connection socket and hand-tighten the screws on the connector.

5 Set-up and connection





It is absolutely essential to comply with the manufacturer's instructions in the separate operating manual of the vacuum sensor!



Fig. 16: Vacuum sensors of different manufacturers



The vacuum sensor comes supplied in a calibrated state.

After the freeze-dryer has been switched on, the vacuum sensor needs several minutes until it is ready for operation.



5.6 Vacuum pump



Please refer to the separate operating manual of the vacuum pump and the exhaust filter!

The vacuum pump must be connected to the vacuum connector of the unit. The electrical connection is located inside the housing next to the vacuum pump.

The oil mist that escapes when the pump is in operation must be retained or carried off by way of an oil mist separator.

- We strongly recommend using an oil mist separator. This filter prevents air pollution by oil mist.
- In order to carry off the oil mist, connect a suitable hose to the exhaust port of the vacuum pump (RZ-2.5 and RC-6: ½", DUO 5 or DUO 10 ¾").
- The hose must be laid so that the condensate cannot flow back into the pump. In the case of upward leading hoses, we recommend using a separator (Woulfe's bottle or wash bottle).

5.7 Pressure control valve

The pressure control valve is integrated in the suction pipe between the vacuum pump and ice condenser chamber. During certain, specified process phases, it interrupts the volume flow to the vacuum pump (see chapter 2.2.1 - "General information on freeze-drying")



The pressure control valve is integrated into the system. Only specialised personnel is authorised to perform maintenance or repairs.

5 Set-up and connection



5.8 Rubber valves

The rubber valves (part no. 121860) enable the connection of round-bottom flasks, wide-neck filter bottles, or distributors for ampoules to a manifold or drying chamber. Depending on the connector of the components, the blue plug can be removed.

- 1 Locking handle
- 2 Aeration connection
- 3 Vessel connection
- 4 Rubber plug
- 5 Connection to freezedryer (e.g. via a manifold)



Fig. 17: Rubber valve



The rubber valves come supplied in an ungreased state. This is why a thin layer of vacuum grease must be applied to the connector of the freezedryer as well as to the vessel connector prior to start-up in order to ensure trouble-free operation.

In position A (see figure below), the aeration connector is open and the vessel connector is closed. The accessory will be aerated while the vacuum inside the drying chamber is maintained. As a result, vessels can be exchanged without any interruption of the drying process.

In position B, the aeration connector is closed and the vessel connector is open. The connected accessory is connected to the freeze-dryer.

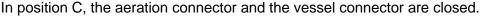




Fig. 18: Possible positions of the locking handle



6 Operation

6.1 Initial start-up



Before the initial start-up, please ensure that your freeze-dryer is properly set up and installed (see chapter 5 - "Set-up and connection")

6.2 Installation of accessories

The accessories must be completed in accordance with the drying method that is applied as well as in accordance with the scope of supply. Please contact our sales department if you have any queries.

6.3 Preparation

The ice condenser chamber must be clean and dry.

- Remove any water residues from the preceding run.
- Close the media drain valve and the aeration valve.
- Ensure that all of the valves of the accessories are closed.
- Switch the vacuum pump on.

6.4 Switching the freeze-dryer on



The freeze-dryer is equipped with a main switch and a mains power switch (see chapter 2.1.1 - "Functional and operating elements"). The main switch establishes the mains power supply and the mains power switch supplies the control system of the freeze-dryer with power.

- Actuate the main switch.
- Actuate the control switch.

The LSCplus control unit performs a self-test and an initialisation. This may take several seconds.

 Follow the safety instructions and hazard warnings (see chapter 3 -"Safety")!



6.5 LSCplus control system

The control system LSCplus (Lyo Screen Control plus) was specifically developed for the control of freeze-drying processes. The clear user interface enables the intuitive operation of the unit.



Fig. 19: Start screen of the LSCplus control unit (example)

6.5.1 User interface

The system is operated via a touch panel, i.e. by touching the buttons on the display. Every button is marked by a frame. Pressing the button activates the associated function. Depending on the function, a dialog box opens, a value can be changed, or a transaction can be confirmed.

Manual Program	Options ?
Standby	2010-10-2 07:22:01
Set values: view/edit Set	Actual
∞ h:m Section time	0:00 h:m Total time
-20.0 °C Shelf	0:00 h:m Section time
	20.0 °c Icecondens.
	1000 mbar Vacuum
	20.0 °C Shelf 1
Operating mode: select/start	Stop

Fig. 20: Buttons are marked by a frame



The user interface is divided into four main windows that can be called up by touching the corresponding buttons:

Manual

This window is also the standard user interface. It is used to control the freeze-drying process manually.

Program

This area is used to create and execute programs for automating the freeze-drying process and for making it reproducible. This function is only available in combination with the programmer module PGMplus option.

Options

This window is used for personalised settings that enable the users to adapt the system as far as possible to their respective area of activity.

?

In this window, the users can find all of the relevant information concerning the control system at a glance. In the event of enquiries at the factory, these data facilitate the assignment and expediting of the processing of the enquiries.

6.5.1.1 Main window "Manual"

This main window shows all of the relevant process data. Here, the individual phases of a freeze-drying process can be controlled manually.



Status line

1

5

- 2 Set process values
- 3 Button "Set values: view/edit"
- 4 Actual values of the current process
 - Button "Tools"
- 6 Button "Schematic system diagram"
- 7 Button "Process- and equipment messages"
- 8 Button "Operating mode: select/start"
- 9 Button "Stop"

Fig. 21: Overview of the main window "Manual"



Status line (1)

This line shows the operating status of the freeze-dryer as well as the active phase.

The status line also shows the current date and time. The clock is batterybuffered and must be reset after a failure (chapter 6.5.1.3 - "Main window "Options "", section "Administration"").

In addition, the drive symbol provides information concerning the status of the external data storage device or of the network drive. The following symbols are possible:

No symbol	No USB storage device or LAN network connected
	USB storage device connected
	Process recording on a USB storage device active
	Network available, but no network drive connected
	Network drive connected (e.g. LPCplus, LyoLogplus)
	Network drive connected and process recording active

Set process values (2)

In the manual mode, the set values for the individual phases of the freezedrying process must be entered prior to the start of the process. Value ranges have been saved for the various phases. These value ranges can be displayed in the input window with the aid of the buttons "min" or "max" (see chapter 6.5.2.1 - "Entering set values in the manual mode").

Button "Set values: view/edit" (3)

This button is used to call up the various parameters that can be edited.



Actual values of the current process (4)

This area shows the current process data. The fields can be configured as desired:

• Select the button of the field that is to be adapted. A dialog box opens:

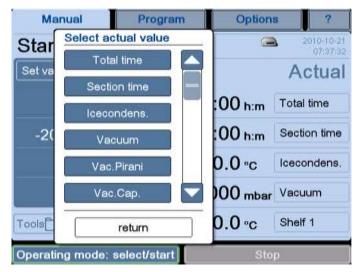


Fig. 22: Dialog box "Select actual value"

• Select the desired configuration or quit the dialog box by pressing the "return" button.

In this way, it is possible to configure a personalised overview of the actual values.

Dialog box "Tools" (5)

This dialog box is used to call up various aids and resources.

Vapour pressure curve for ice and water

A diagram shows the relationship between the pressure and sample temperature. The pressure and temperature values can be changed by pressing the buttons or by moving the arrows (see figure, item 1). The other value will be adapted automatically.

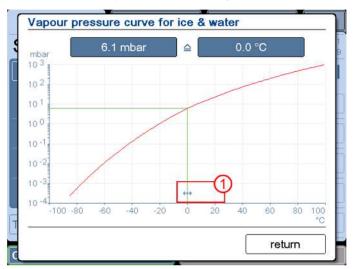


Fig. 23: Dialog box "Vapour pressure curve for ice and water"



Option: USB process recording (see chapter 6.6 - "Optional extensions")

- Select the "Process recording" function in the dialog box "Tools".
- Select the input fields ("Batch data"). A keyboard for the data input will be displayed.
- If necessary, select the "Options" tab, choose between manual or automatic recording, and define a recording interval.
- Press the "return" button in order to close the dialog box.

The process recording will now run in the background.

	Manual	Program	Options	?
St	USB proces	s recording	<u> </u>	2010-10-21
Se	Recording t Recording t File size [Ki	file ime [h:m] Byte]		
	Number			
	Name	2		
	Note			
	Record	ling start	Recording s	top
Toc	Option	าร	return	n]
Ope	rating mode:	select/start	Stop	

Fig. 24: Dialog box "USB process recording"

Option: Pressure increase test (only with the \rightarrow double-chamber method)

The \rightarrow pressure increase test can only be performed when the freeze-dryer is equipped with an intermediate valve. The performance is possible in the manual mode as well as in the program mode. Additionally, the pressure increase test can be automatically performed as part of a program (see chapter 6.5.3.1 - "Creating a program").

- Select "Pressure increase test" in the "Tools" dialog box.
- Enter the set values for the duration and maximum pressure increase with the aid of the buttons.
- Start the pressure increase test. The test time will be displayed. After the end of the test, a status message (pressure increase was successful or failed) will be displayed together with the measured values:



Test not active	
Duration test [sec.] Vacuum [mbar] Vacuum : begin of test [mbar] Vacuum : end of test [mbar] Vacuum : acceptable [mbar] Pressure increase [%]	60 1020 1000 1020 1100 2.0
Duration pressure increase test [sec.]	60
Max. pressure increase [%]	10.0
Test start Ter	st stop
ſ	return

Fig. 25: Dialog box "Pressure increase test"

• The "Test stop" button stops the pressure increase test.

Dialog box "Schematic system diagram" (6)

Pressing the button "Schematic system diagram" displays a schematic diagram of the system on the left-hand side of the screen, including all the components. Active components are displayed in green. Touching a component calls up its name and \rightarrow *reference designator*.



Fig. 26: Schematic system diagram with the name and reference designator of the component



Dialog box "Process and equipment messages" (7)

This dialog box shows and saves all of the error messages and other messages. In the event of an error or message, the window "Process and equipment messages" will open automatically. In addition, a sound signal is emitted until the error is acknowledged.

Malfunctions are divided into three categories:

- Red: error messages
- Orange: process messages
- Yellow: general messages

The representation of the message provides information on its current status. A double frame around a message means that the error has not been eliminated yet. The colour of the button "quit" changes from blue to grey once the message has been acknowledged.



Fig. 27: Representation of an error message

The advantage of this system is that malfunctions that occurred during the night can be discovered the next day even if the cause of the malfunction has already been eliminated.

The dialog box cannot be quit until all of the messages have been acknowledged.

If a message has been acknowledged although the malfunction has not been eliminated, the button "Process and equipment messages" will be displayed in the respective colour of the malfunction in the main window.



1 The malfunction has been eliminated, but the message has not been acknowledged yet

- 2 The malfunction has not be eliminated and the message has not been acknowledged yet
- 3 The malfunction has not been eliminated yet, but the message has been acknowledged

Fig. 28: Dialog box "Process and equipment messages"



<u>Details</u>

Touching the message calls up details concerning the error message:

- Cause of the message
- Effects of the message
- Measures to eliminate the error
- → Reference designator
- Error counter (indicates how often this error has occurred) and the time stamp of the last error message.

Use the arrow keys to call up the individual windows.



The error message text is always followed by an error code. Always indicate the error code in the event of enquiries or service requests!

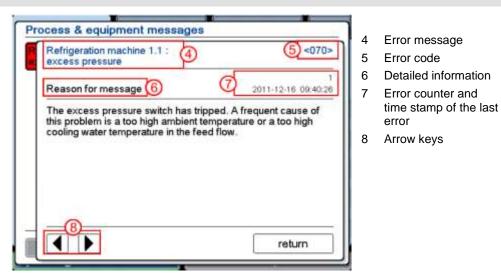


Fig. 29: Details concerning an error message



The texts of the process and error messages are not included in this operating manual.

You can order these documents from our service department.





After the set values have been entered for the process, the process can be started manually with this function (see chapter 6.5.2 - "Manual freeze-drying").

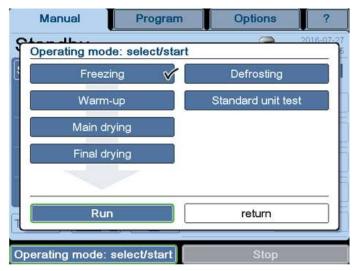


Fig. 30: Dialog box "Operating mode: select/start" (The version of the dialog box that is displayed depends on the equipment of the freeze-dryer.)

Standard unit test

Apart from the process phases of the freeze-drying process ("Freezing", "Warm-up", "Main drying", and "Final drying") and the operating mode "Defrosting", the button "Unit Test" is also available. This button opens a selection of a test with fixed parameters. After consultation with the manufacturer, this test can be performed in order to check the functionality and processes of the freeze-dryer.

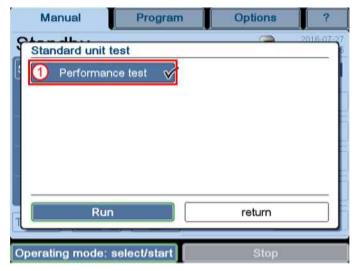


Fig. 31: Dialog box "Standard unit test"



Performance test (1)

The performance test is used to determine the following performance parameters:

- vacuum decrease rate
- final vacuum
- minimum ice condenser temperature
- shelf cooling rate
- minimum shelf temperature
- shelf heating rate



Prior to performing a test, ensure that the chamber is dry and unloaded and that the ice condenser is defrosted.

Procedure:

- In the main window "Manual", select the button "Operating mode: select/start" – "Standard unit test".
- Select "Performance test" and start the test via the "Run" button.

The test will be performed. The parameters will be measured at defined points of time, evaluated, and displayed in a dialog box in the form of a table. The result will be indicated by way of a dialog box.

If the freeze-dryer is equipped with the LPCplus SCADA software, the results will be documented in the event list.

Evaluation		
Ice condenser temperature af	ter 2 h	
Evacuation rate	1,000 mbar 1 mbar	xxx min
	1,000 mbar 0.1 mbar	xxx min
	1,000 mbar 0.01 mbar	xxx min
Final vacuum after 2 h		xxx mbar

Evaluation:

Please contact the Martin Christ Gefriertrocknungsanlagen GmbH for an assessment of the results..

Button "Stop" (9)

Pressing this button stops the current process. The system switches to the standby status.



6.5.1.2 Main window "Program"

In the main window "Program", pre-programmed freeze-drying processes can be loaded and edited and new programs can be created with the PGMplus programmer module. For this function, the PGMplus programmer module must be available.



The PGMplus programmer module is an option that is not included as standard (see chapter 6.5.3 - "Option: freeze-drying with the PGMplus programmer module"). If the programmer module is not enabled, programs can only be created and edited for demonstration purposes.

NOTE

The execution of a program, however, is not possible!

	Manual	Program	Options ?		
	nmary		2 New program	1	Program list Button "New
01. 02. 03. 04. 05.	edit, copy, delete Christ standard Antibodies DMSO Ceramics emuls Nutrient media/t		Load Load Load Load Load	3	Button "New program" Buttons "Load"

Fig. 32: Overview of the main window "Program"

Program list (1)

After the selection of the main window "Program", an overview of the programs that have already been saved will be displayed. Pressing the button "Load" (3) behind the program name calls up the program data. Programs can be loaded, edited, copied, or deleted (see chapter 6.5.3 -"Option: freeze-drying with the PGMplus programmer module").

Dialog box "New program" (2)

In this dialog box, new programs can be created either based on an already existing program or completely from scratch (see chapter 6.5.3.1 -"Creating a program").



6.5.1.3 Main window "Options "

The main window "Options" is used to perform fundamental basic settings of the control system in order to adapt it perfectly to the respective area of application of the freeze-dryer.

General

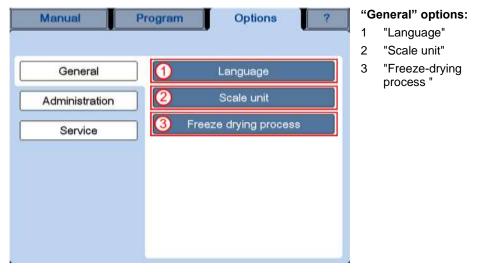


Fig. 33: Overview of the main window "Options/General"

Language (1)

The control system can be used in several languages which can be selected via the dialog box.

ange language		
Deutsch	中文	Türkçe
English 🗸	Português	Hrvatski
Français	Español	Polski
Svenska	Vlaams	Русский
Italiano	Nederlands	Magyar
Accept	1	return

Fig. 34: Dialog box "Change language"



NOTE

Change scale unit (2)

This dialog box is used to change the unit of measurement for the temperature and vacuum.

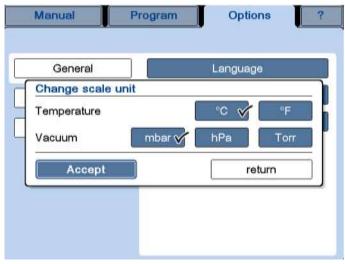


Fig. 35: Dialog box "Change scale unit"

Freeze-drying process (3)

This function depends on the unit type and is not available for all types of freeze-dryers.

Prior to the start of the process, the correct process must be selected. The following processes are available:

- \rightarrow Single-chamber method (inside): drying inside the ice condenser chamber
- → Double-chamber method (outside): drying outside the ice condenser chamber
- Double chamber method LyoCube (outside): drying outside the ice condenser chamber, but with the CHRIST LyoCube (a rectangular drying chamber that can be loaded from the front)

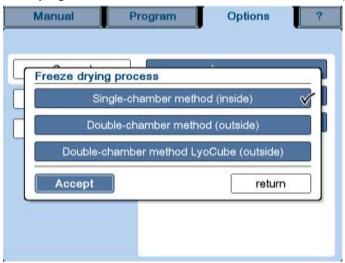


Fig. 36: Dialog box "Freeze-drying processes"



Administration

Manual
General Administration Service

Abb. 37: Dialogfenster "Optionen" / Verwaltung

Date/Time (1)

The LSC_{plus} control system is equipped with an integrated, battery-buffered clock. After a failure of the buffer battery, the date and time must be reset.

General	Date/Time
Change date/	time
Date [y:m:d]	2010 - 10 - 21
Time [h:m]	8:51
Accept	return
	Configuration of sensors

Fig. 38: Dialog box "Change date/time



Network (2)

This dialog box is used to change various system settings.



The modifications will not become effective until after a restart of the unit.

Manu	al	Program		Options	?
G Admi S	IP addres 192 Subnet n 255	. 168 .	ttings 230 224 0	. 70 . 0	
	MAC add	dress 00-21-0		-39 return	ors

Fig. 39: Dialog box "Network"

System properties (3)

This dialog box is used to change various system settings.

System proper	rties	
Beeper		On
Click on touch	1	On
Behaviour of the the case of insu cooling	e pressure control valve in ifficient ice condenser	closed
Accept		return

Fig. 40: Dialog box "System properties"

Beeper: The beeper sounds in the event of a malfunction, for example.

- If the setting is "On", the beeper sounds every few seconds until the user acknowledges the message.
- If the setting is "Silent", the beeper sounds once when the malfunction occurs.
- If the setting is "Off", the beeper will not sound at all.

Click on touch: If this function is active, a clicking sound can be heard whenever the system registers that a button has been touched.



Behaviour of the pressure control valve in the case of insufficient ice condenser cooling: If this function is active (button "closed"), the pressure control valve will close at an ice condenser temperature of \geq -20°C during the drying process in order to avoid damage to the vacuum pump caused by the withdrawal of condensable gases. A corresponding error message will be displayed. Pressing the button again deactivates the function (button "controlled").

Access protection (4)

In this dialog box, the access rights can be managed on several levels and they can be protected with a password.

In the factory setting with an activated access protection, data can be viewed but not edited.

Access protec	tion	
Access protecti	on Off	On s
Password timer	runtime [sec.]	60
Operator passv	vord	change
Maintenance pa	assword	change
Administrator p	assword	change
Accept]	return

Fig. 41: Dialog box "Access protection"

Î NOTE

The other buttons cannot be activated unless the access protection is active.

Password timer runtime: In order to prevent unauthorised access, the system will automatically switch back to the default setting after a predefined period of time.

In this case, there is a small lock symbol in the status line and below this symbol the remaining time until the lock will be active is counted down. At the same time a button with a big lock symbol will be displayed in the actual values field.



Fig. 42: Countdown of the password timer and the button with the lock symbol

٠





The button with the lock symbol blocks the access immediately and the system switches to the default setting.

Manual	Program	Options	?
Standby		6 🛋	2010-10-21 09:06:27
Set values: view/edit	Set	A	ctual
	Freezing		

Fig. 43: Access blocked, the data cannot be edited

User/maintenance/administrator password: For each of these access levels, certain editing rights have been defined. They can be enabled with the corresponding password.

The rights of the various access levels are detailed in the following table.

Action	User	Maintenance	Administrator
Editing of the data of the current process run (e.g. selection of the operating mode, changing of set values)	✓	\checkmark	√
Editing of maintenance functions (e.g. oil change of the vacuum pump)		✓	✓
Editing of the default settings (e.g. editing of the access protection, creating and editing of programs, editing of system settings)			√

Device options (5)

This dialog box lists all of the device options that are available for the unit in question. A list of all the possible options can be found at chapter 6.6 - "Optional extensions". Options that require a series-number-specific release code are marked with the symbol (" $\mathbf{\hat{e}}$ ").

Maj	nual	Progra	m	Options	?	
Ad	 Device Wirel Air in Aera Hot g Conce Prograd Proce Proce Proce LyoC 	e options ess Shelf Teo jection	chnology ule PGMp LYOLOG USB ion LPCp	lus plus		

Fig. 44: Dialog box "Device options" (example)



If the freeze-dryer is to be extended by an option, this option must be enabled via this dialog box.

- Press the button "Add option". An input window opens.
- Enter the six-digit Christ activation code that was supplied for this option. Note that the keys are case sensitive.

Options can be removed in the same way.



The modifications will not become effective until after a restart of the unit.

Configuration of sensors (6)



The sensor configurations depend on the equipment version of the unit. If the configuration is incorrect, the correct operation of the unit cannot be guaranteed.

This dialog box is used to configure the existing vacuum and pressure sensors. For the vacuum as well as pressure, there are two different measuring methods, and for each of these methods, different sensors can be selected.

• In order to change the sensor, press the button with the sensor name. The possible models will be displayed one after the other.

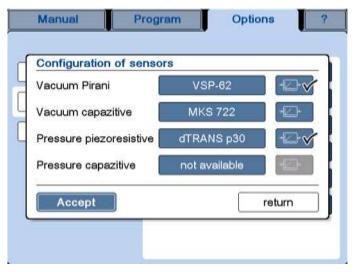


Fig. 45: Dialog box "Configuration of sensors"

The buttons on the right-hand side of the sensors show a control symbol. The tick marks on the buttons indicate the control sensors, i.e. the sensors that are decisive for the vacuum inside the unit. If there is a second sensor, the data of this sensor are simply used for comparison.



Service		
Manual	rogram Options ?	"Service" options:
		1 "Operating hours"
	1	2 "Failure memory"
General	Operating hours	3 "Wireless shelf technology"
Administration	2 Failure memory	4 "Vacuum pump oil change"
Service	3 Wireless Shelf Technology	5 "Maintenance"
	4 Vacuum pump oil change	6 "Sensor adjustment"
	5 Maintenance	
	6 Sensor adjustment	

Fig. 46: Dialog box "Service" (varies depending on the type of system)

Operating hours (1)

This dialog box is used to call up the number of operating hours of the various components of the freeze-dryer, e.g. the refrigeration unit, vacuum pump, or pressure control valve. In addition to the name, the \rightarrow *reference designator* is also displayed.

These data are provided for the purpose of information only. They cannot be edited.

ſ	General Operating hours	Operating hours
1	Refrigerating maschine	1.1
	Operating hours [h:m]	15:01
	Start-up cycles	14
		return

Fig. 47: Dialog box "Operating hours" (here: refrigeration unit 1.1)

Failure memory (2)

The failure memory stores the most recent messages of the process and equipment information system. These messages can be viewed in this dialog box. The failure memory includes the last 32 messages. If this number is exceeded, the oldest message will be overwritten.

Use the arrow keys to call up the individual messages.

The error message text is always followed by an error code.



Î Note

Always indicate the error code in the event of enquiries or service requests!

Failure memory	1/1
Refrigeration machine 1.1 : excess pressure	<070>
Date [y:m:d] Time [h:m]	2012-09-27 07:29:48
	return

Fig. 48: Dialog box "Failure memory"

Wireless shelf technology (3)



This function depends on the unit type and is not available for all types of freeze-dryers.

The \rightarrow wireless shelf technology enables the wireless control and monitoring of the shelf temperature. For this purpose, an address must be assigned to the WST modules of the shelves. The number of available addresses is fixed depending on the unit variant.

Wireless Shelf Te	chnology
Shelf address	1
	Address assignment
8 <u></u>	Version info
	return

Fig. 49: Wireless shelf technology – assignment of shelf addresses

•

6 Operation



- In order to assign an address, open the dialog box "Wireless Shelf Technology".
- Ensure that only the WST module to which an address is to be assigned is connected. If several modules are connected, they will all receive the same address, resulting in a communication conflict.
- Press the button "Address assignment", process the prompt, and confirm the address assignment.

Vacuum pump oil change (4)

The system monitors the oil change interval of the vacuum pump. The interval can be adapted to the vacuum pump model and utilisation. When the end of an oil change interval is reached, a corresponding message will be displayed.

- Acknowledge the message.
- Change the oil of the vacuum pump.
- Reset the operating hour counter in the dialog box "Vacuum pump oil change" by pressing the "reset" button.

acuum pump oil o	hange	
il change interval [h	::m]	500:00
Vacuum Pump 1 Last oil change 20	00-01-01	
	nce last oil change	2:38
		reset
		return

Fig. 50: Dialog box "Vacuum pump oil change"

Maintenance (5)

The maintenance interval of the unit is fixed at 3,000 operating hours or at least one maintenance per year.

When the end of a maintenance interval is reached, a corresponding message will be displayed.

- Acknowledge the message.
- Make an appointment for the maintenance of your freeze-dryer (see section 8 "Maintenance and service").
- After the maintenance, our service engineer will reset the operating hour counter in the dialog box "Maintenance".



CAUTION

6 Operation



Fig. 51: Dialog box "Maintenance"

Sensor adjustment (6)

Misadjusted sensors will lead to incorrect measurement values, which in turn will have a negative effect on the process control.

In this dialog box, the sensors are adjusted in terms of a predefined reference value.

- Press the button in order to select the sensor. A selection menu will be displayed.
- Enter a reference value and press the button "Adjust".

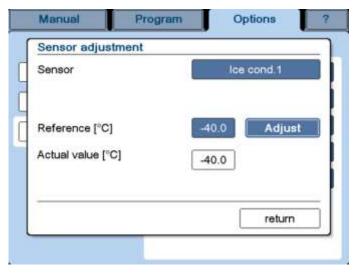


Fig. 52: Dialog box "Sensor adjustment"



6.5.1.4 Main window "?"

This main window includes the most important information concerning your freeze-dryer:



Fig. 53: Freeze-dryer system information, here for Alpha 2-4 LSCplus

In the event of enquiries at the manufacturer, please state the number that is stated here.

6.5.2 Manual freeze-drying

In the manual mode, the user switches manually from one freeze-drying phase to the next. The manual mode is activated by calling up the main window "Manual".



NOTE

Prior to any freeze-drying process, the correct method must be selected (chapter 6.5.1.3 - "Main window "Options "", section "Freeze drying process (3)").

The set values for the individual process phases (freezing, warm-up, main drying, and final drying) are defined prior to the start of the process. Then, the freeze-drying process can be started via the dialog box "Operating mode: select/start".



If the freeze-drying process is to be started directly with the "main drying" (sublimation) phase, the vacuum pump must be warmed up approximately 15 minutes prior to the process start. Failure to do so will result in a corresponding warning message when the process starts.



Î NOTE

If in one section the value " ∞ " (infinite) is selected for a process phase, the next phase must be started manually via the button "Operating mode: select/start".

In the manual mode, the set values of the active phase can be changed during the process run. In this case, the control system adapts the unit to the new set values as quickly as possible.

After the completion of a phase, the system switches to the next phase without switching the unit to standby. The transition from "freezing" to "warm-up" takes place automatically. After the completion of the warm-up phase, this dialog box will be displayed:

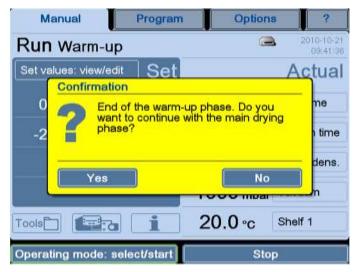


Fig. 54: Dialog box after the completion of the warm-up phase

The unit will remain in the warm-up phase until you confirm.

The transition from "main drying" to "final drying" again takes place automatically. After the completion of the "final drying" phase, the system displays another enquiry with which the freeze-drying process will be terminated. The unit remains in the "run" mode until the enquiry is confirmed.

The process can be stopped any time by pressing the "Stop" button. In this case, the unit will be switched to standby.

6.5.2.1 Entering set values in the manual mode

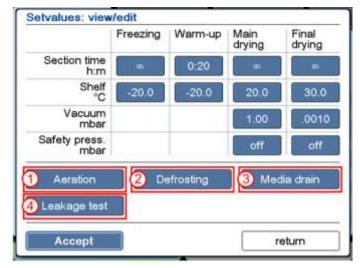
The system has stored set values for every phase, and for every value there are pre-defined value ranges that can be determined in the various dialog boxes by pressing the buttons "min" and "max".

In order to protect the product, $a \rightarrow safety \ pressure$ value can be entered in every drying section.



Viewing or editing the set values:

Press the button "Set values: view/edit" (see chapter 6.5.1.1 - "Main window "Manual""). The following dialog box will be displayed:



- 1 "Aeration" button
- 2 "Defrosting" button
- 3 "Media drain" button (special equipment)
- 4 "Leakage test" button (option)

Fig. 55: Dialog box "Set values: view/edit"

Fields that are displayed in the form of buttons can be edited.

- Aeration (1)
 If the freeze-dryer is equipped with an automatic aeration valve, this button is used to define the aeration pressure.
- Defrosting (2)

This button is used to pre-define the defrosting time and temperature¹. In addition, this button is used to define whether the operating mode "media drain" (see below) will be started automatically after the defrosting process.

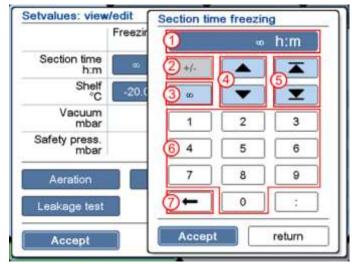
• Media drain (3)

If the freeze-dryer is equipped with an automatic media drain for condensate water or another medium, the opening time of the drain valve can be pre-selected in this dialog box.

Leakage test (4) This function is optional (see chapter 6.6 - "Optional extensions"). This button displays the parameters that are used for the leakage test. In addition, the test time and the chamber volume must be stated (depending on the accessories that are used).

¹ not for hot-water defrosting





Numerical values can be edited with the aid of a numerical keypad:

1 Set value display

- 2 Button for changing the sign (e.g. when entering temperature values)
- 3 "∞" (infinite)
- 4 Button for editing the value in pre-defined steps
- 5 Selection of a possible maximum or minimum value

Input of a value via the numerical keypad

7 Button for deleting the displayed value

Fig. 56: Editing set values

- Confirm the new value and quit the numerical keypad by pressing the button "Accept".
- Confirm the input and quit the dialog box via the button "Accept".
- If the dialog box is closed by the button "return", the changes will be discarded.

6.5.3 Option: freeze-drying with the PGMplus programmer module

Unlike in the manual mode, an entire freeze-drying process can be executed fully automatically and under reproducible conditions with the aid of the PGMplus programmer module.



The PGMplus programmer module is an option that is not available as standard. If the programmer module is **not** enabled, programs can be created and edited for demonstration purposes only. The execution of a program, however, is not possible in this case!

Freeze-drying programs are divided into sections (1). Every section in the program has certain set values (2). A program must include a minimum of two sections, and the maximum number of possible sections is 64. 32 program storage locations are available.

In every program, the system always displays four consecutive sections in order to show their connection.



Manu	lai	Progr	am	Optio	2
01. Christ	standard	8		Suration Sec 2:00 14	tions
1	Load Sec.01	Freezing Sec.02	Sec.03	Sec.04	
2 Time html		1:00	1:00	0.30	Insert section
Sheff *C		-20.0	-20.0	-15.0	Delete section
Vacuum					>> more
Safety press mbar					
AT shelf *C		off	off	off	Copy program
AT product *C		off	off	off	Delete program
LyoControl-Po		off	off	off	Save

Fig. 57: Representation of a freeze-drying program

Program sections

When the programmer module executes a freeze-drying process, it executes the various sections that were created one after the other until the last section is completed.

Within the various sections, the system calculates linear ramps for the temperature and vacuum. These ramps start with the set value of the previous section and end with the set value of the current section.

As a result, there is not abrupt change of the set value from section to section, but a steady adaptation.

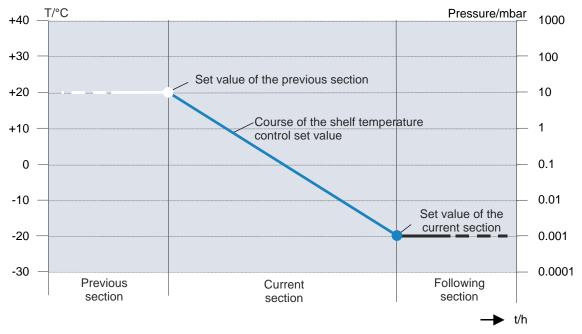


Fig. 58: Graphical representation of the course of the control set value

In order to protect the product, a \rightarrow safety pressure value can be entered in every drying section.



Conditions for switching to the next section

For all of the program sections to be executed automatically, certain switching conditions must be fulfilled at the end of each section. If these conditions are not fulfilled, e.g. due to incorrect set values, a corresponding process message will be displayed and the section will be extended.

<u>Ice condenser temperature</u>: This value is checked only when the system switches from freezing (loading) to drying. The ice condenser temperature must be \leq -40°C.

<u>Vacuum</u>: This value is checked only during the drying phase. The actual vacuum can differ from the set value by 20% maximum. In the case of a set value of 0.001 mbar (final vacuum), there will be no check. In order to reach the vacuum as quickly as possible, a section time of 1 minute can be preselected. Since this is not possible in practice, a process message will be issued for the first time after 15 minutes in this case.

 ΔT shelf: This value defines the permissible deviation of the shelf temperature from the set value. At the end of the section, the actual temperature of the shelves (in the case of WST shelf 1) will be compared to the set value. If the shelf temperature is beyond the permissible range, the section will be extended until the deviation is within the permissible range.

 ΔT product: This value defines the permissible deviation of the product temperature (measured by product sensor 1) from the set value. During the freezing phase, the product temperature may exceed the set value by the defined value. In the drying phase, the system will provide a signal to the user if the actual value lies below the set value by more than the permissible deviation.

<u>LyoControl-Rx</u> (option with LyoControl LCplus): The value LyoRx defines the minimum permissible value of the LyoControl sensors (measured by the LyoControl sensor 1) during the drying phase. If the actual value falls below this limit, the shelf heater will be switched off in order to prevent the product from thawing due to excessive energy input by the shelf temperature control system. The LyoControl value is checked only during the main and final drying phases.

<u>Ap pressure increase test (option)</u>: Depending on the selected mode, the pressure increase test can also be used as a condition for switching to the next section (see chapter 6.5.3.1 - "Creating a program", option: pressure increase test). In the last drying section, two pressure increase tests will be performed and evaluated. If both values are not greater than the specified "pressure increase" parameter, the condition for switching to the next section is fulfilled. If this is not the case, the section will be extended. If the actual value exceeds the set value before the preselected time has elapsed, the test will be aborted in order to prevent the product from thawing.

 ΔT comparative (option): This value indicates the difference between a Pirani sensor and a capacitive vacuum sensor in per cent based on the actual value of the capacitive vacuum sensor. When the sublimation rate decreases, the difference decreases as well. As a result, the value is an indicator for determining the drying end of the main draining phase. If the set value is not reached by the end of the main drying phase, the section will be extended.



Vacuum pump warm-up

Apart from the execution of the various sections, the PGMplus programmer module also controls other tasks that need to be performed during the process run, e.g. the automatic activation of the vacuum pump.

If the pressure control valve is closed, the PGMplus programmer module shifts the warm-up phase of the vacuum pump to the freezing phase so that it is **before** the first drying section. Since the pressure control valve remains closed during the warm-up of the vacuum pump, neither the freezing phase nor the pressure inside the drying chamber will be affected.

The duration of the warm-up phase can be specified separately for every program (see chapter 6.5.3.1 - "Creating a program").

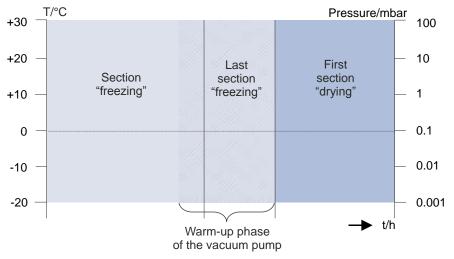


Fig. 59: The warm-up phase of the vacuum pump is shifted to the freezing phase directly before the first drying section



6.5.3.1 Creating a program

To create a new program:

 Press the button "New program" in the main window "Program". The dialog box that is displayed offers various different program templates. The scroll bar on the right-hand side can be used to scroll through the list.



- 1 Button for creating a blank program
- 2 Scroll bar for selecting a program template

Fig. 60: Dialog box "Select program template"

Creating a blank program (1)

This button is used to call up a blank program template. Only section 1 is pre-defined as the loading section. In this phase, the start conditions of the programs are defined. The room temperature (20°C) is the standard default.



If the product is frozen outside the freeze-dryer (double-chamber method), the shelf temperature must be adapted accordingly in section 1.

For all the other sections, the set values must be defined (see figure below):

- Press the button "Insert section" (4) and select the position of the section as well as the freeze-drying phase. The section will be inserted at the defined position.
- Adapt the parameters of the inserted section. All of the values are not available in all of the freeze-drying phases.
- Insert and edit the next section.



During the creation of a program, the order of the individual freeze-drying phases must be maintained. This means that it is not possible to insert a freezing section after a main drying section.



Manu	al	Progr	am 🕧	Option	9 ?		rogram" buttons:
06. test1				uton Section	<u>a</u>	1	"Program"
	Load Sec.01	Freezing Sec.02	Main drying Sec.03	J 3	•	2 3	"Program name" "Show diagram "
Time htm		1:00	1:00	4	Insert section	4 5	"Insert section " "Delete section "
Shelf *C	20.0	20.0	20.0	6	Delete section	6	Button for calling u
Vacuum mbar			10.0	6	>> more	1	set values
Safety press. mbar			off			7	"Save"
∆T sheff °C		off	off		Copy program		
ΔT product *C		off	off		Delete program		
LyoControl-Rx		off	off	0	Save		

Fig. 61: Editing a blank program template

- The button "Delete section" (5) can be used to delete sections.
- The button ">>more" (6) can be used to enter more functions and set values in a program-related manner, depending on the type of freezedryer (see the sections below).
- Proceed in this manner in order to create an entire program according to your specific needs.
- Pressing the button "Diagram" (3) displays the program in the form of a diagram.
- Pressing the button "Program name" (2) calls up an input field in which the name of the program can be changed.
- The button "Save" (7) can be used in between or at the end of the program creation in order to save the program.
- The button "Program" (1) calls up the main window. The system will ask the user whether the program shall be saved if this has not been done yet.

The program will be automatically assigned to the first free program storage location. The creation of the program is now complete and it can be loaded.

Button ">>more"

The button ">>more" can be used to enter more functions and set values in a program-related manner.



	Additional functions and set values	2
01. C	Vacuum pump warm-up [min] 15	
	Pressure increase test	
	Comparative Pressure Measurement	ction
		ction
Ň		re
Safety		
4		gram
ΔΤ		gram
LyoCor	return	

Fig. 62: Selection of program-related functions and set values

Vacuum pump warm-up See chapter 6.6 - "Optional extensions"

Option: Pressure increase test (only with the \rightarrow double chamber method) (see chapter 6.6 - "Optional extensions")

The \rightarrow pressure increase test can only be performed when the freeze-dryer is equipped with an intermediate valve.



The indication of the measurement value "dp Test" can be configured in chapter 6.5.1.1 - "Main window "Manual"" under "Actual values of the current process" in the dialog box "Select actual value".

In contrast to the pressure increase test in the manual mode, repeated pressing of the button in the program mode enables the selection of different variants.

Periodic pressure increase test:

The test will be performed periodically during the entire main or final drying phase. The parameters "Duration test" and "Time between tests" apply. The maximum pressure increase is limited to 100%, referring to the actual value at the beginning of the measurement. If the value is exceeded, the pressure increase test will be aborted in order to prevent the product from thawing.

• Progress condition:

In the last main drying or final drying section, two pressure increase tests will be performed and evaluated. The start point will be calculated automatically by the control system. The parameters "Duration test" and "Time between tests" apply.

If both tests have been passed, the drying process will be considered complete at the current conditions. Thus the progress condition for switching to the next section is fulfilled and the next section will be initiated.

Otherwise, a process message will be issued, the current section will be extended, and further pressure increase tests will be executed



periodically until the condition is fulfilled. If the actual value exceeds the set value before the preselected time has elapsed, the test will be aborted in order to prevent the product from thawing. If the value is exceeded, the pressure increase test will be aborted in order to prevent the product from thawing.

• Periodic & progress condition:

This variant is a combination of the possibilities that are described above. The test will be performed periodically during the entire main or final drying phase. The maximum pressure increase is limited to 100%, referring to the actual value at the beginning of the measurement. If this value is exceeded before the test time has elapsed, the particular test will be aborted in order to prevent the product from thawing. If the last two tests have been passed (amount of pressure increase below the limit "Pressure increase"), the drying process will be considered complete at the current conditions. Thus the progress condition for switching to the next section is fulfilled and the next section will be initiated.

Otherwise, a process message will be issued, the current section will be extended, and further pressure increase tests will be executed periodically until the condition is fulfilled.

Disabled:

No pressure increase test will be executed during the main or final drying phase. There will be no evaluation.

alues
3
ndition
30
10
5.0
return

Fig. 63: Dialog box "Pressure increase test" in the program mode



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Option: Comparative pressure measurement (see chapter 6.6 - "Optional extensions")



The indication of the measurement value "dp Comp" can be configured in chapter 6.5.1.1 - "Main window "Manual"" under "Actual values of the current process" in the dialog box "Select actual value".

The comparative pressure measurement can be activated or deactivated by pressing the button in the dialog box.

• Progress condition:

If the difference between the readings of the Pirani and the capacitive vacuum sensor at the end of the last main drying section is below the limit " Δp Comparative", the drying process will be considered complete at the current conditions. Thus the progress condition for switching to the next section is fulfilled and the next section will be initiated. Otherwise, a process message will be issued, the current section will be extended, and the current section will be extended until the condition is fulfilled.

• Disabled:

There will be no comparative pressure measurement during the main drying phase. There will be no evaluation.

Progres	ss condition
p Comparative [%]	15.0

Fig. 64: Dialog box "Comparative pressure measurement"

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Program templates (2)

The PGMplus programmer module offers 14 different program templates that include recipes for all kinds of freeze-drying applications. They are suggestions for orientation purposes and must be adapted to the specific area of application of the freeze-dryer.

Following the selection of a program template, a window containing information concerning the drying process will be displayed.

	Manual	Program	Options	?
S o o	Attention - sugge is required. • Recipe for pro- chamber).	ient media/bacteri estion for orientation cess A (drying inside	n! Further optimisa e the ice condense	ər
0	(please refer to concerning the	the ice condenser the operating instru- temperature range	uctions for informa of the system).	ition
٥		ozen separately. In f ce condenser cham		

Fig. 65: Information concerning the program template (here: a recipe for nutrient media/bacteria)

After the conformation of the information, the program template will be displayed.

• Similar to a blank program, sections can be added or deleted and the set values can be adapted accordingly.

6.5.3.2 Editing a program

An existing program can be modified as long as it has not been loaded.

- Select the program to be edited from the list in the main window "Program".
- Press the button with the program name in order to call up a window that displays the program data.
- Perform the desired modifications and save the program (see chapter 6.5.3.1 "Creating a program").
- Close the dialog box by pressing the "Program" button.

The program has now been changed in the existing program storage location.



It is possible to switch to the manual mode during a program run, e.g. in order to edit a program during the runtime. The point of time for continuing the program run can be defined by selecting the desired start section and a start time.



6.5.3.3 Copying a program

If a new program is to be created based on an already existing program, the already existing program can be copied. A free program storage location must be available for this purpose.

- Select the program to be copied from the list in the main window "Program".
- Press the button with the program name in order to call up a window with the program data.
- Press the button "Copy program" in order to create a copy of the existing program.
- Edit and save the copy (see chapter 6.5.3.1 "Creating a program").
- Close the dialog box by pressing the "Program" button.

The program will be automatically assigned to the first free program storage location.

6.5.3.4 Loading a program

If a freeze-drying process is to be executed and controlled by a program, the program must be loaded.

- Call up the main window "Program". This window includes a list of all the programs that are saved.
- Press the button "Load" behind the program name. A dialog box will be displayed in which the start section, the start time or the start temperature can be adapted to any specific needs. The "Info" button can be used to call up a brief description of the active program, including information concerning the remaining runtime and the end of the program. It also enables a graphical representation of the process sequence.

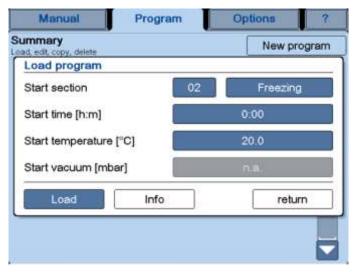


Fig. 66: Dialog box "Load program"

- Press the button "Load" in order to accept the program data. The display switches to the standard user interface.
- Press the button "Program start" in order to start the freeze-drying process.



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Fig. 67: The freeze-drying process can be started with the aid of the button "Program start".

- During the freeze-drying process, the description of the active program can also be called up from the main window "Program" and via the button "Info".
- When the "Stop" button is pressed, the freeze-dryer switches to the standby manual freezing mode.



The program starts with section 02 "Freezing" by default. If, however, section 01 "Load" is to be used as the starting point of the program, this section must be selected manually. In this case, the button "Program continue" will be displayed after the start of the program. When the precooling (loading) process is complete, this button must be pressed in order to continue with the program.

6.5.3.5 Deleting a program

The PGMplus programmer module offers 32 program storage locations. If they are all occupied, a program must be deleted before a new one can be created.

- Select the program to be deleted from the list in the main window "Program".
- Press the button with the program name in order to call up a window with the program data.
- Press the button "Delete program". The system will then display a dialog box with an enquiry.
- Following the confirmation of the enquiry, the program will be deleted.

The program storage location on the list is now empty and the number is not shown on the list.



6.6 **Optional extensions**

Comparative pressure measurement

see also chapter 6.5.3.1 - "Creating a program", button ">>more" During the sublimation, i.e. when the concentration of water vapour molecules is rather high in the atmosphere, the value that is provided by the gas-type-dependent vacuum sensor of the "Pirani" type (e.g. Thyracont VSP62/63) deviates from the value that is provided by a capacitive vacuum sensor (e.g. MKS 722B). When the proportion of water vapour molecules decreases towards the end of the main drying phase, the two sensors fall increasingly in line with one another. This difference will be evaluated and used as an indicator for identifying the drying end of the main drying phase.

Leakage test

see also chapter 6.5.3.1 - "Creating a program"

The leakage test enables the chamber of the freeze-dryer to be tested for tightness in view of any gaseous or liquid media. Since absolutely tight components simply do not exist, a leak rate is determined. The parameters for the leakage test have been developed by Martin Christ Gefrier-trocknungsanlagen specifically for freeze-dryers. In a first step, these parameters (vacuum, ice condenser temperature) must be reached. It is not until the conditions are fulfilled that the pressure control valve closes. Then, the actual leakage test is performed in a second step. The leak rate that is calculated after the end of the test provides information concerning the tightness of the system.

PGMplus programmer module

see also chapter 6.5.2.1 - "Entering set values in the manual mode" The PGMplus programmer module enables the fully automatic execution of an entire freeze-drying process under reproducible conditions.

Pressure increase test

see also chapter 6.5.3 - "Option: freeze-drying with the PGMplus programmer module", button ">>more"

The pressure increase test can only be carried out with the \rightarrow doublechamber method. During the pressure increase test, the intermediate valve prevents the flow of steam from the drying chamber to the ice condenser so that the water vapour of the \rightarrow sublimation cannot flow off. The result is a more or less distinct pressure increase that is measured in the product chamber. The pressure increase test is used as a criterion for the automatic switching between main drying and final drying and also for identifying the end of the process.

USB process recorder

see also chapter 6.5.2 - "Manual freeze-drying", dialog box "Tools" This feature enables the recording of a running process on a USB storage medium. After the end of the process recording, the process data can be viewed on the PC with LyoLogplus and be printed. It is also possible to import the data directly into an Excel file.

6 Operation



Lyo Control measuring system

The Lyo Control measuring system can be used to determine the crystallisation state of the product. In the liquid state, the electrical resistance is very low. During the freezing process, the resistance increases. The LyoRx control sensor measures the electrical resistance.

LyoLogplus data logging software

LyoLogplus is a data logging software program by Martin Christ Gefriertrocknungsanlagen GmbH that is specifically adapted to the requirements of freeze-drying processes. Apart from the graphical representation of the measurement data of currently running processes, it also enables the data export for additional evaluation.

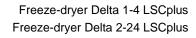
LPCplus SCADA system

The Christ LPCplus system consists of the <u>Supervisory Control And Data</u> <u>Acquision (SCADA) software program by Martin Christ Gefriertrocknungs-</u> anlagen GmbH and a dedicated PC. The system is connected to the LSCplus controller of the freeze-dryer via Ethernet LAN and provides the operation of all of the freeze-drying functions as well as process recording (measurement data and process events), process documentation and data backup. Furthermore, it enables the comfortable administration of freezedrying programs/recipes and users.

6.7 Switching the freeze-dryer OFF

The freeze-dryer must be in the standby status.

- Switch the control system off by pressing the control switch.
- Pressing the main switch disconnects the freeze-dryer from the power.



Malfunctions are displayed in the dialog box "Process & equipment messages" (see chapter 7.2 - "Process and error messages"). An acoustic signal sounds when an error message is generated.

- Eliminate the source of the problem (see the following chapter).
- Acknowledge the error message.

7.1 General malfunctions

Type of error	Possible reason	Correction
No indication on the display	 No power in the mains supply (see chapter 7.1.1 - "Power failure"). Power cord is not plugged in. Fuses have tripped. The control switch and/or the main switch is set to off. 	 Check the mains power supply fuse. Plug in the power cord correctly. Check the on-site fuses. Switch the control switch and the main switch on.
The touchpanel does not react at all or it does not react correctly	• The sensitivity of the touchpanel is misadjusted.	Contact the service department (see chapter 7.3 - "Service contact")
The password input fails	The password is not correct.	 Inform the administrator. If you have lost the administrator password: contact the service department (see chapter 7.3 - "Service contact")
Insufficient vacuum	 Incorrect connection of the small flange connection(s). 	• Loosen the connection. Place the centring ring with the inner sealing ring in a centred manner between the flange connections and connect it with the clamping ring. Ensure that the centring ring neither slips out of place nor gets jammed.
	Dirty or damaged lid or door seal.	Clean the lid or door seal and replace it if necessary.
	• The ground-in stopper of the attached drying chamber is not installed correctly.	• Grease the ground-in stopper evenly and over the entire sealing surface with vacuum grease.
Leakage in the media drain valve	 The media drain valve is soiled with drying residues or wool particles from cleaning cloths. The O-rings are worn. 	 Clean the media drain valve (see chapter 8.1.3 - "Aeration valve, media drain valve") and replace it if necessary. Replace the O-rings.
Leakage in a rubber valve	The valve is soiled.	Check the valves individually (see chapter 7.1.2.4 - "Rubber valves")



Type of error	Possible reason	Correction
The displayed vacuum value is not correct	Incorrect calibration	 Calibrate the vacuum sensor (see the separate operating instructions of the vacuum sensor).
	• The vacuum sensor is soiled	Clean the vacuum sensor.
	(e.g. due to water residues).	Check the vacuum display with
	 The vacuum sensor is defective. 	the aid of a reference device (if available).
		see chapter 7.1.2.5 - "Vacuum sensor"
The vacuum pump is not activated	 See the separate operating instructions of the vacuum pump. 	 See the separate operating instructions of the vacuum pump.
Insufficient ice condenser or shelf temperature	• The overpressure switch of the refrigeration unit has tripped.	• Let the unit cool down.
	The thermal circuit breaker has tripped.	• Ensure sufficient air circulation (see chapter 7.1.3 - "Insufficient ice condenser temperature")



If it is impossible to eliminate the errors, contact the Christ service department!

7.1.1 Power failure

The control system continues with the process after a power failure. The preselected conditions remain saved even during a process run.

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.

- In the final drying phase, the product has reached a residual moisture content of approx. 5%. Below this value, the product is generally not damaged even if the power failure lasts for a longer period of time.
- If the product is in the main drying phase, we recommend aerating the unit, removing the product, and storing it in a deep-freeze. The defrosted condensate must be drained off prior to the next start.



7.1.2 Insufficient vacuum



The vacuum checks must be carried out when the ice condenser is frozen.

7.1.2.1 Small flange connections

Leakages are often due to improper small flange connections between the various components and hose connections or to leakages in the valves.

- Loosen the connection and place the centring ring (with sealing ring inside) in a centred manner between the flange connections.
- Seal the connection with the clamping ring by tightening the wing nut.
- Ensure that the centring ring neither slips out of place nor gets jammed.



Fig. 68: Small flange and centring ring



Fig. 69: Small flange with centring ring and small flange



Fig. 70: Attaching the clamping ring



Fig. 71: Tightened clamping ring



7.1.2.2 Aeration valve, media drain valve

A malfunction of the aeration valve or the media drain valve may have several causes. One potential source are contaminants such as product residues within the valve.

- Switch the freeze-dryer off and disconnect the mains plug.
- Clean the valve (see chapter 8.1.3 "Aeration valve, media drain valve").
- Put the freeze-dryer into operation again.

If there is still a leakage, the freeze-dryer must be checked by qualified specialist personnel (see chapter 7.3 - "Service contact").

7.1.2.3 Pressure control valve

A malfunction of the pressure control valve may have several causes.



The inspection of the valve must be carried out by qualified specialist personnel (see chapter 7.3 - "Service contact").

7.1.2.4 Rubber valves

In order to identify a leaking rubber valve, the valves must be checked individually:

- Remove the rubber valve and seal the connection at the drying chamber with a rubber stopper.
- Check the tightness under vacuum until the leaking valve has been localised.
- Clean the valve or replace it if necessary.

7.1.2.5 Vacuum sensor

Vacuum sensors have a limited service life and can be ordered as spare parts (e.g. VCP 63, order no. 312011). Functional test:

 Connect the vacuum sensor directly to the suction side connector of the vacuum pump.

If a final pressure of at least 0.011 mbar is reached (with a vacuum pump that has reached its operating temperature), the sensor and the vacuum pump are OK.



7.1.3 Insufficient ice condenser temperature



Ensure sufficient ventilation. Do not place any paper, cloth, or similar material behind or under the unit, since otherwise the air circulation will be impaired.

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.

In these cases, the refrigeration unit will be switched off automatically. If the permissible operating conditions are re-established after a cool-down phase of several minutes, the refrigeration unit will be switched on again automatically.

The malfunctions are displayed in the process and equipment information window.

The minimum ice condenser temperature of approx. -55° C or approx. -85° C (depending on the type of freeze-dryer) is reached when the ice condenser is not loaded and the ice condenser chamber is evacuated.

7.2 Process and error messages

The **LSCplus** control system displays the complete process and error messages (see chapter 6.5.1.1 - "Main window "Manual"", dialog box "Process and equipment messages"), which is why they are not included in this operating manual.

You can order these documents from our service department.



7.3 Service contact

In the event of queries, malfunctions, or spare part enquiries:

From Germany:

Contact Martin Christ Gefriertrocknungsanlagen GmbH An der Unteren Söse 50 37520 Osterode (Germany) Tel. +49 (0) 55 22 / 50 07-44 44 E-mail: <u>support.lab@martinchrist.de</u>

Outside Germany:

Contact our agency in your country. All agencies are listed at <u>www.martinchrist.de</u> \rightarrow [Sales Partners]



If you would like to utilise our after-sales-service, please state the type of your freeze-dryer and its serial number.



8 Maintenance and service

The freeze-dryer and the accessories are subject to high mechanical stress. Thorough maintenance performed by the user extends the service life and prevents premature failure.



If corrosion or other damage occurs due to improper care, the manufacturer cannot be held liable or subject to any warranty claims.

- Use soap water or other water-soluble, mild cleaning agents for cleaning the freeze-dryer and the accessories.
- Do not use corrosive and aggressive substances.
- Do not use solvents.
- Do not use agents with abrasive particles.
- Do not expose the freeze-dryer or its accessories to intensive UV radiation (e.g. sunlight) or thermal stress (e.g. by heat generators).
- Do not turn the unit upside down in order to clean it.

8.1 Maintenance

8.1.1 General

The general state of the freeze-dryer must be checked at regular intervals. Any defects must be eliminated immediately! The following points are of particular importance:

- dirt
- leaks
- corrosion
- bent system components
- loose screw and flange connections
- higher noise levels
- loose cables
- open cable ducts
- missing or illegible safety notes and hazard warnings
- missing or illegible inscriptions on components, pipes (direction of flow) and cables
- etc.

8 Maintenance and service



Cleaning of the freeze-dryer

- Switch the freeze-dryer off by actuating the mains power switch and disconnect the power cord from the wall outlet before cleaning.
- If the freeze-dryer has been contaminated with toxic, radioactive, or pathogenic substances, clean the inside immediately with a suitable decontamination agent (depending on the type of contamination, see chapter 8.2 - "Disinfection of the drying chamber and accessories").
- Remove product residues thoroughly with a cloth.
- Open the lid/drying chamber when the freeze-dryer is not in use so moisture can evaporate.

8.1.2 Ice condenser chamber

Before each start-up, ensure that the ice condenser chamber is free from water residues.

- Open the media drain valve to drain off any liquid. Then, close the valve.
- If necessary, wipe the ice condenser chamber dry with a cloth.

8.1.3 Aeration valve, media drain valve

Contaminants such as product residues may lead to an insufficient vacuum. In this case, the aeration valve and the media drain valve must be cleaned.

- Switch the freeze-dryer off and disconnect the mains plug.
- Remove the valve core.
- Clean the valve core and the opening with a moist cloth.
- Clean the O-rings and inspect them for any damage. Damaged O-rings must be replaced.
- 1 Valve opening
- 2 Valve core
- 3 O-rings

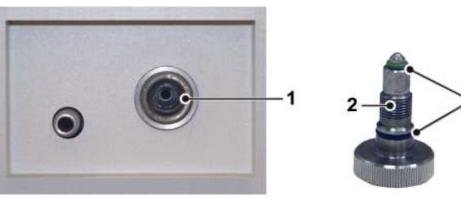


Fig. 72: Valve opening and valve core with O-rings (example, varies depending of the type of freeze-dryer)

- Reinsert the valve core.
- Put the freeze-dryer into operation again.

If the vacuum is still insufficient, the freeze-dryer must be checked by qualified specialist personnel (see chapter 7.1.2.2 - "Aeration valve, media drain valve").



8.1.4 Heat exchanger (only for air-cooled freeze-dryers)

A lamellar heat exchanger is used for cooling the refrigerant that is compressed by the refrigeration unit. This air-cooled heat exchanger is located at the back of the unit (see chapter 2.1.1 - "Functional and operating elements").

Dust and dirt impair the cooling effect of the air flow. Dust on the lamellas prevents the exchange of heat and, thereby, impairs the performance and power of the refrigeration unit. Strong soiling may cause the unit to fail.

This is why the selected set-up location should be as clean as possible.

- Check the heat exchanger at least once per month for soiling and clean it if necessary.
- Please contact the Christ service department if you have any queries (see chapter 7.3 "Service contact").

8.1.5 Vacuum pump



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

The stress of the vacuum pump in conjunction with a freeze-dryer is usually not very high. This is why the recommendations in this operating manual may differ from the information that is provided by the pump manufacturers. Under normal operating conditions, the following maintenance tasks concerning the vacuum pump must be performed at regular intervals:

- Check the oil level of the vacuum pump once per week. If necessary, top it up with oil.
- Check the running pump for any unusual noise.
- Ensure that the pump has reached its operating temperature prior to changing the oil.
- Perform the first oil change after approximately 100 operating hours.
- The other oil changes depend on the operating conditions. In general, an interval of 500 to 1,000 operating hours is sufficient.
- Please contact the Christ service department if you have any queries (see chapter 7.3 "Service contact").

8 Maintenance and service



8.1.6 Exhaust filter (oil mist separator)



Please refer to the separate operating manual of the vacuum pump and the exhaust filter!

The oil mist that is emitted by the vacuum pump in quantities that depend on the working pressure must be led to the outside or to an exhaust hood or similar. If this is not possible, the pump must be equipped with an exhaust filter (oil mist separator).

- Observe the liquid level in the collecting vessel of the filter.
- Remove the condensate in time (please refer to the information provided by the manufacturer in the separate operating manual).

8.1.7 Vacuum sensor



Please refer to the separate operating manual of the vacuum sensor!

The vacuum sensor has only a limited service life. Especially carboncontaining substances, e.g. alcoholic compounds, reduce the service life extremely.

- The vacuum sensor is maintenance-free.
- Remove any soiling on the outside with a cloth.

8.1.8 Accessories



For the care of the accessories, special safety measures must be considered as these are measures that will ensure operational safety at the same time.

Chemical reactions as well as stress-corrosion (combination of oscillating pressure and chemical reaction) can affect or destroy the metal and plastic parts. Barely detectable cracks on the surface can expand and weaken the material without any visible signs.



- Check the material regularly (at least once a month) for
 - cracks
 - visible damage of the surface
 - pressure marks
 - signs of corrosion
 - other changes.
- Replace any damaged components immediately for your own safety.
- Immediately rinse off the accessories if any liquids that may cause corrosion come into contact with them.
- Clean the accessories outside the freeze-dryer once a week or preferably after each use.

8.2 Disinfection of the drying chamber and accessories

- Use commercially-available disinfectants such as, for example, Incidur[®], Meliseptol[®], Sagrotan[®], Buraton[®], or Terralin[®] (available at specialised trade).
- The freeze-dryers and the accessories consist of various materials. A possible incompatibility must be considered.
- Before using cleaning or decontamination agents that were not recommended by us, contact the manufacturer to ensure that such a procedure will not damage the freeze-dryer.
- Please contact us if you have any queries (see chapter 7.3 "Service contact").



If dangerous materials (e.g. infectious and pathogenic substances) are used, the freeze-dryer and accessories must be disinfected.

8 Maintenance and service



8.3 Service



In the event of service work that requires the removal of the panels, there is a risk of electric shock or mechanical injury. Only qualified specialist personnel is authorised to perform this service work.

The freeze-dryer is subject to high mechanical stress. In order to be able to withstand this high level of stress, high-quality components were used during the production of the freeze-dryer. Nevertheless, wear cannot be excluded and it may not be visible from the outside.

This is why we recommend having the freeze-dryer checked by the manufacturer during an inspection once per year.

Information and appointments:

From Germany:

Contact

Martin Christ Gefriertrocknungsanlagen GmbH An der Unteren Söse 50 37520 Osterode (Germany) Tel. +49 (0) 55 22 / 50 07-44 44 E-mail: support.lab@martinchrist.de

Outside Germany:

Contact our agency in your country. All agencies are listed at <u>www.martinchrist.de</u> \rightarrow [Sales Partners]



If you would like to utilise our after-sales-service, please state the type of your freeze-dryer and its serial number.

8.4 Return of defective parts

Although we exercise great care during the production of our products, it may be necessary to return a unit or accessory to the manufacturer.

In order to ensure the quick and economical processing of returns of freeze-dryers, rotational vacuum concentrators, spare parts, or accessories, we require complete and extensive information concerning the process. Please fill in the following forms completely, sign them, enclose them with the return package, and send them together with the product to:

Martin Christ Gefriertrocknungsanlagen GmbH An der Unteren Söse 50 37520 Osterode (Germany)



8 Maintenance and service

1. Declaration of decontamination

As a certified company and due to the legal regulations for the protection of our employees and of the environment, we are obliged to certify the harmlessness of all incoming goods. For this purpose, we require a declaration of decontamination.

- The form must be filled in completely and signed by authorised and specialised personnel only.
- Affix the original form in a clearly visible manner to the outside of the packaging.



We will return the part/unit if no declaration of decontamination is provided!

2. Form for the return of defective parts

This form is for the product-related data. They facilitate the assignment, and they enable the quick processing of the return. If several parts are returned together in one packaging, please enclose a separate problem description for every defective part.

- A detailed problem description is necessary in order to perform the repair quickly and economically.
- Upon request, we will prepare and submit to you a cost estimate prior to performing the repair. Please confirm such cost estimate within 14 days. If the cost estimate has still not been confirmed after 4 weeks, we will return the defective part/unit. Please note that you must bear the incurred costs.



The part/unit must be packaged in a transport-safe manner. Please use the original packaging for the unit, if at all possible. If the product is dispatched to us in unsuitable packaging, you will be charged the cost for returning it to you in new packaging.

The forms can be downloaded online from <u>www.martinchrist.de</u> \rightarrow [Service] \rightarrow [Overhaul, repair and leak testing].

9 Disposal



9 Disposal

9.1 Disposal of the freeze-dryer

Martin Christ Gefriertrocknungsanlagen GmbH is a registered manufacturer of electric and electronic devices that are solely intended for commercial use.

• Comply with all local rules and regulations.

9.2 Disposal of the packaging

- Dispose of the packaging, after having separated the individual materials.
- Comply with all local rules and regulations.



10 Technical data

Manufacturer:	Martin Christ Gefriertrocknungsanlag An der Unteren Söse 50 37520 Osterode Germany	en GmbH
Туре:	Delta 1-24 LSCplus	Delta 2-24 LSCplus
Part number:	102300	102301
Performance data	Delta 1-24 LSCplus	Delta 2-24 LSCplus
Ice condenser - capacity: - performance: - temperature: - chamber volume:	24 kg max 18 kg / 24 h max approx. –55°C approx. 45 l	24 kg max 18 kg / 24 h max approx. –85°C approx. 45 l
Temperature of the ice condenser during hot-gas defrosting:	approx. 60°C	approx.60°C
Possible shelf temperatures (→ single chamber method): freezing and drying inside the ice condenser chamber	approx. –40°C to +60°C	approx. –50°C to +60°C
Possible shelf temperatures (→ double chamber method): drying outside the ice condenser chamber	Room temperature to +60°C	Room temperature to +60°C
 Max. shelf surface area (→ single chamber method): drying inside the ice condenser chamber drying in injection vials with 	10 shelves, \emptyset 200 mm each A _{total} =0.31 m ² 4 shelves, \emptyset 250 mm each	10 shelves, \emptyset 200 mm each A _{total} =0.31 m ² 4 shelves, \emptyset 250 mm each
sealing under vacuum or nitrogen atmosphere inside the ice condenser chamber	A _{total} =0.18 m ²	A _{total} =0.18 m ²
Max. shelf surface area (→ double chamber method): - drying outside the ice condenser chamber	A _{total} =1.1m ²	10 shelves, Ø 375 mm each A _{total} =1.1m ²
- drying in injection vials with sealing under vacuum or nitrogen atmosphere outside the ice condenser chamber	4 shelves, \varnothing 250 mm each A _{total} =0.18 m ²	4 shelves, \varnothing 250 mm each A _{total} =0.18 m ²
 drying in round bottom flasks or wide-neck filter bottles, max. number 	12 pieces, 24 pieces, or 36 pieces	12 pieces, 24 pieces, or 36 pieces

10 Technical data



Connection requirements (without vacuum pump and accessories)	Delta 1-24 LSCplus	Delta 2-24 LSCplus		
Electrical connection:	3 x 400 V / 50 Hz	3 x 400 V / 50 Hz		
Protection class:	I	1		
IP protection category according to DIN 60529:	11	11		
Apparent power:	0.9 kVA	1.55 kVA		
Nominal current:	3.5 A	3.0 A		
Mains fuse:	10 A F	12 A F		
Vacuum pump connection:	Depending on the type of vacuum pump	Depending on the type of vacuum pump		
Filling quantity:	Delta 1-24 LSCplus	Delta 2-24 LSCplus		
Refrigerant: filling quantity / CO ₂ equivalent - R290: - R404A: - R508B:	 860 g / 3.25 t 34 g / 0.46 t	10 g / < 0.01 t 570 g / 2.15 t 150 g / 2.01 t		
Physical data (without vacuum pump and accessories)	Delta 1-24 LSCplus	Delta 2-24 LSCplus		
Dimensions - Height: - Width: - Depth:	1,090 mm 860 mm 610 mm + 80 mm vacuum connection	1,090 mm 860 mm 610 mm + 80 mm vacuum connection		
Weight:	approx. 215 kg	approx. 250 kg		
Noise level according to DIN 45635:	54 dB(A)	54 dB(A)		
EMC according to EN 55011:	Class B	Class B		
Heat emission:	0.9 kW min. 1.9 kW max.	1.7 kW min. 3.0 kW max.		
Equipment connections:	Delta 1-24 LSCplus	Delta 2-24 LSCplus		
Vacuum connection:	Small flange connection DN25KF (ISO 28403, DIN 2861)	Small flange connection DN25KF (ISO 28403, DIN 2861)		
Condensate drain valve:	Hose nozzle DN10 (outside diameter 12 mm)	Hose nozzle DN10 (outside diameter 12 mm)		
Aeration valve:	Hose nozzle DN6 (outside diameter 10 mm max.)	Hose nozzle DN6 (outside diameter 10 mm max.)		
Vacuum sensor:	SUB D-9 VSP 63	SUB D-9 VSP 63		
Data interface (LAN):	RJ 45	RJ 45		



10 Technical data

Special equipment: water cooling system	Delta 1-24 LSCplus	Delta 2-24 LSCplus
Part number:	102302	102303
Cooling water consumption:	max. 0.23 m ³ /h	max. 0.37 m ³ /h
Heat carried off via the cooling water:	1.9 kW	3.0 kW
Cooling water feed flow connection:	R3/4" with hose nozzle DN13	R3/4" with hose nozzle DN13
Cooling water return flow connection:	R3/4" with hose nozzle DN13	R3/4" with hose nozzle DN13

10.1 Ambient conditions

- The figures are valid for an ambient temperature of +20°C.
- Allowable ambient temperature +10 °C to +25 °C.
- Max. humidity 85% (non-condensing) at +25°C.

10.2 Technical documentation

The technical documentation of this freeze-dryer (e.g. circuit diagram, cooling system) and the safety data sheets of the manufacturers of refrigerant and heat transfer medium is not attached to this operating manual.

You can order these documents from our service department.



11 Appendix

11.1 Mathematical relations

The automatic processes in the "Programmer module" menu (see chapter 6.5.3 - "Option: freeze-drying with the PGMplus programmer module") are based on the following considerations:

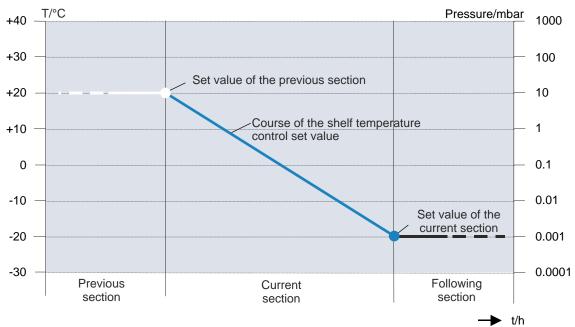


Fig. 73: Graphical representation of the course of the control set value

Calculation of the control set value and of the gradient for the temperature:

 $gradient = \frac{set \ value \ of \ current \ sec \ tion - set \ value \ of \ previous \ sec \ tion}{sec \ tion}$ [°C/min] section time of current section

control set value = set value of previous section + elapsed section time \cdot gradient [°C]

Example:	Section	Set values			
		Section time [h:min]	Temperature [°C]		
	Preceding		30		
	Current	1:00	60		

gradient = $\frac{60^{\circ}C - 30^{\circ}C}{60\min} = \frac{30^{\circ}C}{60\min} = 0.5 \text{ °C/min}$

After an elapsed section time of 30 minutes, for example, the control set value for the temperature is:

Control set $value_{(t=30\min)} = 30^{\circ}C + 30\min \cdot 0.5^{\circ}C/\min = 45^{\circ}C$

Calculation of the control set value for the vacuum:

 $LOG10(set value prev. sect.) + \left(\frac{LOG10(set value current sect. - LOG10(set value prev. sect.))}{section time of current section}\right) + elapsed section time$ [mbar]



11.2 Brief operating instructions

Functional and operating elements:

- 1 LSCplus user interface (see chapter 6.5.1 -"User interface")
- 2 Vacuum pump
- 3 Oil mist separator



Fig. 74: Front of the freeze-dryer

4 Touchpanel



Fig. 75: User interface with touchpanel

- 5 Contact bolt
- 6 Pipe connection of the vacuum pump (behind the cover plate)
- 7 Ice condenser
- 8 Vacuum sensor

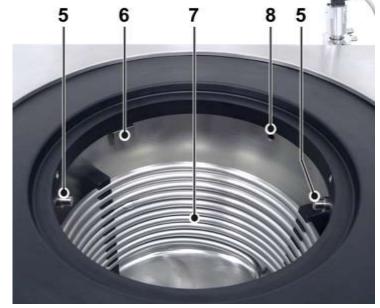


Fig. 76: Ice condenser chamber

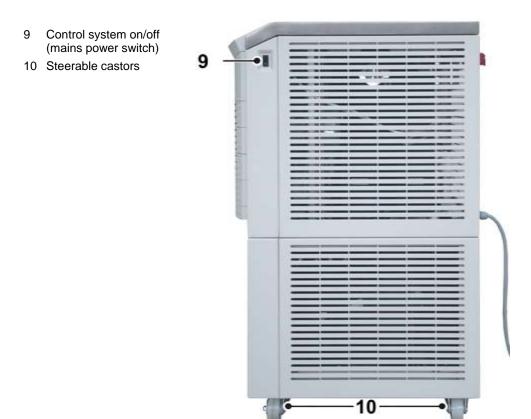


Fig. 77: Right side of the freeze-dryer





- 11 Aeration valve
- 12 Media drain valve
- 13 Fixed castors

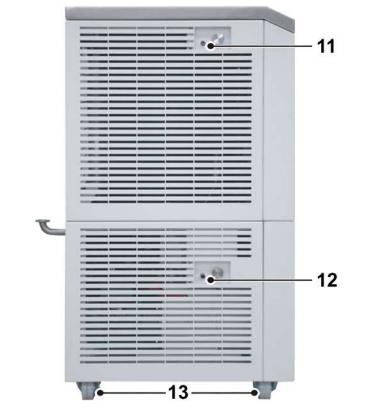


Fig. 78: Left side of the freeze-dryer

- 14 Vacuum sensor
- 15 Mains voltage (Main switch)
- 16 Electrical connection of the vacuum sensor
- 17 Serial interface
- 18 Mains cable
- 19 Heat exchanger of the refrigeration unit (behind the panel)
- 20 Option: Connection for electrical lifting device
- 21 Name plate (see chapter 2.1.2 - "Name plate")
- 22 Vacuum connection

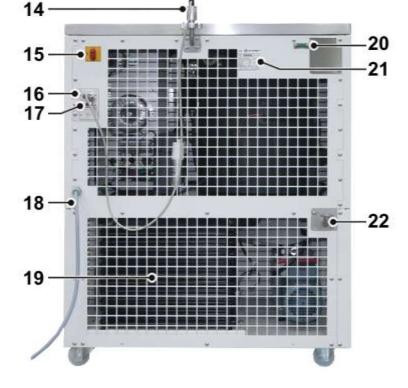


Fig. 79: Rear side of the freeze-dryer

- 1 Status line
- 2 Set process values
- 3 Button "Set values: view/edit"
- 4 Actual values of the current process
- 5 Button "Tools"
- 6 Button "Schematic system diagram"
- 7 Button "Process and equipment messages"
- 8 Button "Operating mode: select/start"
- 9 Button "Stop"



Fig. 80: LSCplus user interface

Step-by-step instructions - shelf drying

1 Freeze the sample separately, e.g. in a deep-freeze.



Ensure that the layer thickness of 1 to 2 cm is not exceeded, since otherwise the drying time needs to be extended.

- 2 Check the ice condenser chamber and ensure that it is completely free from water residues.
- 3 Close the condensate drain valve and install the base plate.
- 4 Switch the unit on 20 to 30 minutes prior to the start of the drying process in order to let the vacuum pump warm up.
- 5 Place the plate rack on the base plate.
- 6 Transport the frozen samples as quickly as possible from the deepfreeze to the freeze-dryer and place them on the shelves.



<u>Recommendation:</u> Store the product vessels on the aluminium shelves or, if possible, the entire rack with the shelves in the deep- freeze. The advantage is that due to the higher cold storage capacity of the aluminium material, the product will remain frozen for a longer period of time so that the sample will not thaw.

- 7 Install the drying chamber. Prior to doing so, check whether the O-ring is completely free of dirt particles. The ground-in stopper of the acrylic glass bell must be greased with high-vacuum grease.
- 8 Ensure that all of the valves of the acrylic glass bell are closed.
- 9 Ensure that the aeration valve is closed.
- 10 Ensure that the condensate drain valve is closed.

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11 Start the main drying process either by opening the manual shut-off valve or by waiting for the electromagnetic valve to open. Vacuum is applied to the chamber and the freeze-drying process commences.



The vacuum pump always runs with maximum power. With this type of freeze-dryer, the power of the vacuum pump cannot be controlled.

- 12 The operating panel displays the vacuum, the ice condenser temperature, and the current operating mode.
- 13 The end of the process is reached when the ice condenser is no longer loaded and when it again reaches a final temperature of approximately -50°C to -54°C. The pressure decreases as a function of the ice condenser temperature.
- 14 Switch the vacuum pump off and aerate the drying chamber via the condensate drain valve or via a rubber valve.
- 15 Switch the unit off by actuating the mains power switch and take the product out of the freeze-dryer.
- 16 Switch the unit on again and start the defrosting process (button "Operating mode: select/start" "Defrosting").



Ensure that no water gets into the pipe connection of the vacuum pump or vacuum sensor.

- 17 Drain the defrosting water via the condensate drain valve on the lefthand side of the unit. To do so, connect a hose to the hose connector and collect the defrosting water in a suitable vessel.
- 18 Keep the freeze-dryer open (i.e. without the lid or drying chamber) when it is not in use so that moisture can evaporate. This increases the service life of the vacuum sensor.

Step-by-step instructions – drying in a flask

1 Freeze the sample separately, e.g. in a deep-freeze.



Ensure that the layer thickness of 1 to 2 cm is not exceeded, since otherwise the drying time needs to be extended.

- 2 Check the ice condenser chamber and ensure that is completely free from water residues.
- 3 Install the drying chamber. Prior to doing so, check whether the O-ring is completely free of dirt particles. The ground-in stopper of the acrylic glass bell must be greased with high-vacuum grease.

11 Appendix	
	 4 Ensure that all of the valves are closed. 5 Let the vacuum pump warm up 20 to 30 minutes before the freeze- drying processes commences.
	6 Connect a frozen sample to a valve.
CAUTION	After the pressure has fallen below 1.030 mbar, a frozen sample can be connected to a valve. The next frozen sample cannot be connected to another valve until the pressure is again lower than 1.030 bar.
Î NOTE	The vacuum pump always runs with maximum power. With this type of freeze-dryer, the power of the vacuum pump cannot be controlled.
	 7 The operating panel displays the vacuum, the ice condenser temperature, and the current operating mode. 8 The end of the process is reached when the ice condenser is no longer loaded and when it again reaches a final temperature of approximately - 50°C to -54°C. The pressure decreases as a function of the ice condenser temperature.
Î NOTE	The drying time depends on the layer thickness of the sample, the solids content of the sample, and the amount of heat that is supplied during the drying process. In the case of a layer thickness of 1 cm, the freeze-drying process usually takes 24 hours.
	 9 Switch the vacuum pump off and aerate the drying chamber via the condensate drain valve or via a rubber valve. 10 Switch the unit off by actuating the mains power switch and take the product out of the freeze-dryer. 11 Switch the unit on again and start the defrosting process (button "Operating mode: select/start" – "Defrosting").
CAUTION	Ensure that no water gets into the pipe connection of the vacuum pump or vacuum sensor.
	 12 Drain the defrosting water via the defrosting water valve on the left-hand side of the unit. To do so, connect a hose to the hose connector and collect the defrosting water in a suitable vessel. 13 Keep the freeze-dryer open (i.e. without the lid or drying chamber) when it is not in use so that moisture can evaporate. This increases the service life of the vacuum sensor.

CHRIST



11.3 EC declaration of conformity in accordance with the EC Machinery Directive

	CHRIST
	e
EC – DECLA	RATION OF CONFORMITY
in accordance with the EC Mac	hinery Directive 2006/42/EC, annex II, part 1, section A
he product named hereinafter was	developed, designed, and manufactured in compliance with
	ety and health requirements of the listed EC directives.
	re not authorised by us or if the product is used in a manner
that is not in line with the inte	ended purpose, this declaration will be rendered void.
Product name:	Freeze-dryer
Product type:	Delta 1-24 LSCplus
	Delta 2-24 LSCplus
Order number:	102300, 102302, 102304, 102306,
oradi namoti.	102308, 102310, 102316, 102318, 102320, 102322
	102320, 102322 102305, 102307,
	102309, 102311, 102317, 102319, 102321, 102323
Directives:	2006/42/EG Machinery Directive
	2014/35/EU Low Voltage Directive
	2014/30/EU EMC Directive
Martin Christ Gefriertrocknu	ngsanlagen GmbH
An der Unteren Söse 50	Authorised representative
37520 Osterode Germany	for CE matters: S. Krippendorff
Osterode, 18/02/2016	
(8:-1	
M. Christ, Management	





11.4 Declaration of conformity – China RoHS 2

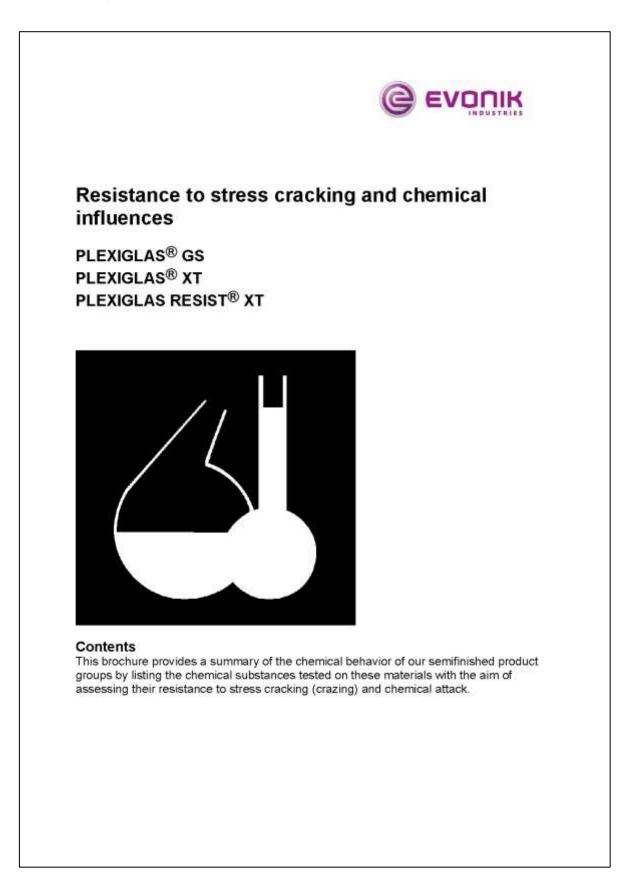
				\mathbf{O}		RIST
	DE	CLARAT	TON OF	CONFOR	RMITY	
China Ro	oHS 2 (Administrative Measures for the Restriction of the Use of Hazardous					
	Sul	bstances in E	lectrical and	Electronic Pro	ducts)	
Alpha 2-4 LSC	plus, Beta 1	-8 LDplus, Be	ta 2-8 LDplus,	olus, Alpha 2-4 Beta 1-8 LSC 24 LSCplus, D	olus, Beta 2-8	LSCplus,
				Dplus, RVC 2-1 h infrared heat		-resistant, RVC
			has made rea reeze-dryers a	sonable effort t ind RVC).	o avoid the us	e of
Mercury and it Lead (Pb) and	ls compound	is: 0.1 %		n Value limit) a I) and its comp promium (Cr (V	ounds: 0.01 %	
Polybrominate	d biphenyls	(PBB): 0.1 %		ed diphenyl eth	39)	23
Polybrominate		表1	Polybrominate	ed diphenyl eth	ers (PBDE): 0	23
Polybrominate		表1	Polybrominate 产品中有害物质 tent of hazardou	ed diphenyl eth 的名称及含量 s substances in t (蒂物质	ers (PBDE): 0	23
		表1	Polybrominate 产品中有害物质 tent of hazardou	ed diphenyl eth 的名称及含量 s substances in t	ers (PBDE): 0	23
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部件名称 Component part (PCA) Electronic PC8,	Table 1 铅 Lead (Pb)	表1 : Name and con 兼 Mercury (Hg)	Polybrominate 产品中有害物质 tent of hazardou 有 Hazardo 餐 Cadmium (Cd)	ed diphenyl eth 的名称及含量 s substances in t r害物质 us substance 大价格 Hexavalent Chromium (Cr (VI))	eers (PBDE): 0 the product 多溴联苯 Poly- brominated biphenyls (PBB)	₹ 求 浪 二 茶 歴 Polybromi- nated diphenyl ethers (PBDE)
部件名称 Component part (PCA) Electronic PCB, cables	Table 1 铅 Lead (Pb) X ¹⁾	表1 : Name and con 兼 Mercury (Hg) O	Polybrominate 产品中有害物质 tent of hazardou Hazardo (Cd)	ed diphenyl eth 的名称及含量 s substances in f (事物质 us substance 六价格 Hexavalent Chromium (Cr (VI))	eers (PBDE): 0 the product 多溴联苯 Poly- brominated biphenyls (PBB)	1% 家澳二苯醛 Polybromi- nated diphenyl ethers (PBDE) O
部件名称 Component part (PCA) Electronic PCB, cables Display Housing Base, metal,	Table 1 辩 Lead (Pb)	表1 : Name and con 兼 Mercury (Hg) 0 0	Polybrominate 产品中有害物质 itent of hazardou Hazardoo 《Cadmium (Cd) 0 0	ed diphenyl eth 的名称及含量 s substances in t 常物质 us substance 大价格 Hexavalent Chromium (Cr (VI))	eers (PBDE): 0 the product 参溴联苯 Poly- brominated biphenayls (PBB) 0 0	1% 求復二苯種 Polybromi- nated diphenyl ethers (PBDE) 0 0 0
部件名称 Component part (PCA) Electronic PCB, cables Display Housing	Table 1 월 Lead (Pb) X ¹⁾ O	表1 : Name and con 兼 Mercury (Hg) 0 0 0 0	Polybrominate Pale paramoniate The paramoniate The paramoniate Pale pale pale pale pale pale pale pale p	ed diphenyl eth 的名称及含量 s substances in t (書物质 us substance 大价格 Hexavalent Chromium (Cr (VI)) 0 0 0 0	eers (PBDE): 0 the product 参溴联苯 Poly- brominated biphenyls (PBB) 0	1% 多復二苯種 Polybromi- nated diphenyl ethers (PBDE) 0 0
部件名称 Component part (PCA) Electronic PCB, cables Display Housing Base, metal,	Table 1 辩 Lead (Pb)	表1 : Name and con # Mercury (Hg) 0 0 0 0 0 0 0	Polybrominate 产品中有害物质 itent of hazardou Hazardoo 《Cadmium (Cd) 0 0	ed diphenyl eth 的名称及含量 s substances in f (事物质 us substances in f (事物质 us substance 大价格 Hexavalent Chromium (Cr (VI))	eers (PBDE): 0 the product 参溴联苯 Poly- brominated biphenayls (PBB) 0 0	1% 求復二苯種 Polybromi- nated diphenyl ethers (PBDE) 0 0 0



	© CHRIST
0:	表示该有害物质在该部件所有均质材料中的含量均在GB/T 26572规定的限量要求以下。 Indicates that the content of the harmful substance in all homogeneous materials of the component part is below the limit as defined in GB/T 26572.)
X:	表示该有害物质至少在该部件的某一均质材料中的含量超出GB-T 26572规定的限量要求。(全业可在此处。根据实际情况对上表打"×"的技术取因进行进一步说明。) Indicates that the content of the harmful substance in at least one homogeneous material of the component part exceeds the limit as defined in GB/T 26752. (Contact the manufacturer for further technical information according to the actual situation.)
9	Contains parts in compliance with exemptions 6c, 7c.I, 7c.II and 37 of 2011/65/EU RoHS.
2)	Contains parts in compliance with exemptions 6a, 6b and 6c of 2011/65/EU RoHS.
	art from the exemptions given in this table, none of the substances listed above have been entionally added to the product or metallic coatings.
Ì	terode, 01/08/2016 ueral Manager



11.5 Resistance to stress cracking and chemical influences "Plexiglas"





Remarks

Brief remarks on the resistance to chemicals other than those listed here, some of them branded products, are made in our leaflet entitled "Chemical resistance of PLEXIGLAS[®] GS and XT" (Ref. No. 211-1).

The physical properties are described in our Product Description leaflets which your stockist holds available for each group of semifinished material.

When using our products you are advised to observe

- the regional Building Regulations and emission laws,
- the applicable standards
- the product liability to VOB (= Contracting rules for award of public works contracts) and BGB (= Civil Code)

• the guidelines of the employers' liability insurance association and others.

Please consult our current sales ranges to see which semifinished products are available in the market.

Contents	Page
1 Introduction 1.1 Chemical resistance 1.2 Resistance to crazing	(varies according to
2 Test results 2.1 Explanation of symbols	computer and printer settings)
2.2 Listing of results	

1 Introduction

On many occasions, the first question to be asked before choosing PLEXIGLAS[®] for a particular purpose is whether they are resistant to specific substances or materials. The answer to this question then decides on their use or non-use.

This is normally tested under standard conditions in the laboratory, on the one hand to evaluate the effect of different agents and, on the other hand, to compare the effect of these on different plastics, e.g. $PLEXIGLAS^{\textcircled{R}}$.



1.1 Chemical resistance

The simplest method for investigating such effects consists in bringing the substance concerned into contact with a specimen without applying any additional load, i.e. by immersing the specimen in a liquid or placing a solid substance on its surface. In this context we speak of testing chemical resistance or insensitivity to staining.

Assessment criteria are the changes in appearance, weight and strength after storage. Exposure period, temperature and concentration of the substance in contact with the material have a pronounced influence on results. In order to obtain reliable information, one would have to simulate the conditions in practical use - time, temperature and concentration - most accurately. This effort, however, is only justified in exceptional cases. In order to reduce test periods to a minimum, we increase the test temperature and/or the concentration. In doing so, we rely on our experience that chemical reactions are accelerated at increasing temperatures.

Tests of this type are described in German standard DIN 53 476, 'Determination of the behavior towards liquids' (Fig. 1).

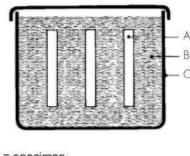
A test period between 1 day, 1 week and 1 month is stated as the time span within which the first changes became visible in the specimen. Short-term testing within 1 minute is performed to identify particularly aggressive substances.

Different types of PLEXIGLAS[®] show certain variations in chemical resistance. Owing to its increased molecular weight, PLEXIGLAS[®] GS is somewhat more resistant than PLEXIGLAS[®] XT or items injection-moulded from PLEXIGLAS[®] moulding compound. This difference, however, is often very slight, so that the resistance lists for these materials are largely identical.

For more precise information on the chemical resistance of the different grades of PLEXIGLAS® see "2.1 Explanation of symbols."

The test results for chemical resistance apply in particular to permanent exposure of stress-free plastics to the agents mentioned.

Fig. 1: Testing of the chemical resistance to DIN 53 476



A = specimen B = agent C = container



1.2 Stress cracking (crazing)

Stress provoked by machining, for example, by thermoforming, screwed fastening, riveting, cold curving or local variations in thermal load, must be allowed for in many fields of application. This stress has to be taken into account when evaluating the behavior of PLEXIGLAS[®].

Where plastics exposed to air are stressed or strained beyond a specific limit, they will sooner (high stress/strain) or later (low stress/strain) develop crazes. Simultaneous exposure to certain agents may drastically reduce the time span up to the onset of crazing. This phenomenon is termed "environmental stress cracking" or just "crazing."

As can be shown by a simple test, only tensile stress causes cracking: if we bend a PLEXIGLAS[®] rod between our hands (Fig. 2) and moisten the stressed convex surface with ethyl alcohol, cracks develop within a short time. The same test on the concave lower surface subjected to compressive stress does not cause crazing even after a long time.

 $\mathsf{PLEXIGLAS}^{\textcircled{R}}$ lends itself to various crazing tests, all of them being fairly demanding as far the preparation and number of specimens, test procedures and testing equipment are concerned.

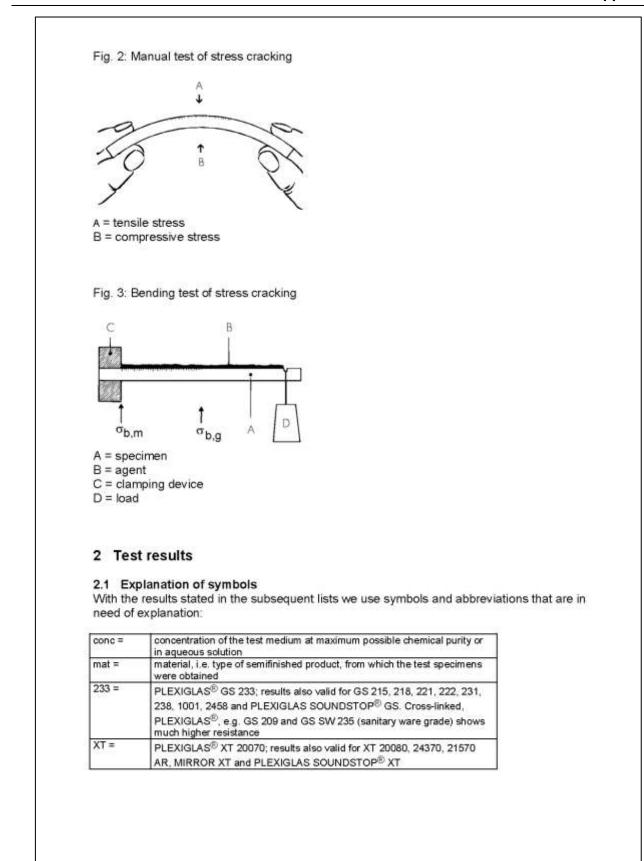
A further difficulty consists in transferring test results to practical conditions, since many users do not have the necessary experience.

A much simpler test method, the so-called **"bending test,"** has been successfully used in our company for over 30 years. The stress conditions it simulates are between those of the tensile creep test and the bending strip method according to DIN 53 499.

The surface of a horizontal test bar, which is held on one side only (Fig. 3), is coated with the test medium and loaded at its free end in such a way that a tensile stress $\sigma_{b,m}$ of no more than 30 MPa is generated near the clamping device. This value decreases linearly towards the loaded end, where it reaches zero. A defined tensile stress is assigned to each point along the surface of the test bar. Crazing sets in at the point of maximum tensile stress and progresses within the test period towards the loaded end, up to a certain point. After a test period of 24 hours at a temperature of 23 °C, the bar is visually inspected for crack propagation. A flexural stress at conventional deflection $\sigma_{b,g}$ is calculated for the end point of crazing.

Long-term experience has shown that products which do not develop crazes after 24 hours at a flexural stress of over 25 MPa and a temperature of 23 °C (and/or at over 15 MPa and a temperature of 50 °C) are not prone to stress cracking in practical use, provided our handling instructions are duly observed.







XT-R =	PLEXIGLAS RESIST [®] XT 41; results also valid for RESIST XT 31 and RESIST XT 21. All RESIST XT grades are more sensitive to chemicals but less prone to crazing than non-modified XT grades.
	Colored PLEXIGLAS $^{\mbox{\scriptsize B}}$ can be expected to behave like the corresponding clear (basic) grades.
RC =	resistance to crazing (Röhm test method 'bending test')
CR =	chemical resistance (similar to DIN 53 476)
EP =	exposure period to the chemical in days; one minute in short-term tests
OE =	overall evaluation, i.e. critical summary of the visual inspections for crazing behavior and chemical resistance

+ = resistant

o = limited resistance

- = not resistant

2.2 Listing of results

Alcohol, mono- and polyhydric

Chemical	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term	OE
Conc						test	
1-Butanol							
100%	233	-	-	28	crazing, swelling	no change	-
100%	хт	-	-	7	pronounced swelling, whitening	no change	-
100%	XT-R	-	-	1	softening, whitening, pronounced swelling	no change	-
1-Hexyl alcohol							
98%	233	-	+	28	no change	no change	0
98%	ХТ	-	o	28	very slight swelling	no change	-
98%	XT-R	-	-	7	swelling, whitening, dulling	no change	-
1-Methoxy-2-propyl alcohol					-		
99%	233	-	-	1	pronounced swelling, chemical attack	no change	-
99%	хт	-	-	7	pronounced chemical attack	no change	-
99%	XT-R	-	-	1	specimens dissolved	surface haze	-
n-amyl alcohol							
100%	233	-	o	28	crazing, swelling	no change	-
100%	ХТ	-	-	28	haze, swelling	no change	1-
100%	XT-R	-	-	1	softening, whitening, pronounced swelling	no change	-
Isopropyl alcohol							
100%	233	-	-	7	swelling, crazing	no change	1-
100%	ХТ	-	-	7	swelling, whitening, crazing	no change	-
100%	XT-R	-	-	1	swelling, whitening, dulling	no change	-



Cyclohexanol							
99,5%	233	-	+	28	no change	no change	0
99,5%	XT	-	+	28	no change	no change	0
99,5%	XT-R	-	-	7	swelling, whitening, dulling	no change	-
Ethyl alcohol							
100%	233	-	-	7	softening, swelling	no change	-
100%	XT	-	-	1	swelling	no change	-
100%	XT-R	-	-	1	softening, swelling, whitening	no change	-
50%	233	-	-	7	swelling	no change	-
50%	ХТ	-	-	1	swelling	no change	-
50%	XT-R	+	-	1	swelling, whitening, dulling	no change	-
Ethylene glycol							
100%	233	-	+	28	no change	no change	0
100%	XT	-	+	28	no change	no change	0
100%	XT-R	-	+	28	no change	no change	0
Ethylene glycol (antifreeze)							
50%	233	+	+	28	no change	no change	+
50%	XT	+	+	28	no change	no change	+
50%	XT-R	+	0	28	slight haze	no change	0
Glycerol							
98%	233	+	+	28	no change	no change	+
98%	XT	+	+	28	no change	no change	+
98%	XT-R	+	+	28	no change	no change	+
Methyl alcohol							
100%	233	-	-	1	softening, swelling	no change	-
100%	XT	-	-	1	softening, swelling	no change	-
100%	XT-R	-	-	1	softening swelling, whitening	slight haze	-
Phenol (dissolved in water)							
5%	233	-	-	1	whitening, tackiness, swelling	no change	-
5%	хт	-	-	1	whitening, tackiness, swelling	no change	-
5%	XT-R	-	-	1	whitening, tackiness, swelling	no change	-

Organic solvents, fuels

Chemical Conc	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term test	OE
Butyl acetate							
99%	233	-	-	1	pronounced swelling, chemical attack	no change	-
99%	хт	-	-	7	pronounced chemical attack	no change	-
99%	XT-R	-	-	1	specimens dissolved	swelling, attack, whitening	-



Acetic ether							
(ethyl acetate)							
99%	233	-	-	1	pronounced swelling, chemical attack	no change	-
99%	хт	-	-	1	pronounced chemical attack	surface slightly dull	-
99%	XT-R	-	-	1	specimens dissolved	swelling, chemical attack, dulling	-
Pentyl acetate (amyl acetate)							
98%	233	-	-	28	swelling, chemical attack	no change	-
98%	хт	-	-	28	pronounced chemical attack	no change	-
98%	XT-R	-	-	1	specimens dissolved	slight chemical attack, dulling	-
Acetone							
99%	233	-	-	28	pronounced swelling, chemical attack	no change	-
99%	хт	-	-	1	specimens dissolved	slight chemical attack, slight dulling	-
99%	XT-R	-	-	1	specimens dissolved	swelling, chemical attack, whitening	-
Cyclohexanone						g	
99%	233	-	-	7	specimens severely attacked	no change	-
99%	хт	-	-	28	pronounced chemical attack	no change	-
99%	XT-R	-	-	1	specimens dissolved	dull surface	-
Diethyl ketone							
99%	233	-	-	1	pronounced swelling, chemical attack	no change	-
99%	хт	-	-	1	pronounced chemical attack	slight chemical attack, slight dulling	-
99%	XT-R	-	-	1	specimens dissolved	swelling, chemical attack, whitening	-
Ethyl methyl ketone			_				
99,5%	233	-	-	1	pronounced swelling, chemical attack	no change	-
99,5%	хт	-	-	1	pronounced chemical attack	slight chemical attack, slight dulling	-
99,5%	XT-R	-	-	1	specimens dissolved	swelling, chemical attack, whitening	-
Cyclohexane							
99,5%	233	-	+	28	no change	no change	0
99,5%	XT	-	+	28	no change	no change	0
99,5% Isooctane	XT-R	-	-	28	swelling, whitening	no change	-
99,5%	233	-	+	28	no change	no change	0
99,5%	ХТ	-	+	28	no change	no change	0
99,5%	XT-R		0	28	slight haze	no change	+



n-Heptane							
99%	233	-	+	28	no change	no change	0
99%	ХТ	-	+	28	no change	no change	0
99%	XT-R	-	-	28	swelling, colour change to opaque white	no change	-
n-Hexan							
99%	233	-	+	28	no change	no change	0
99%	ХТ	-	+	28	no change	no change	0
99%	XT-R	-	-	28	swelling, whitening	no change	-
Formamide							
99%	233	-	+	28	no change	no change	0
99%	ХТ	-	+	28	no change	no change	0
99%	XT-R	-	+	28	no change	no change	0
n-Methylformamide							
99%	233	-	-	7	swelling, haze	no change	-
99%	хт	-	-	1	swelling, chemical attack, whitening	no change	-
99%	XT-R	-	-	1	swelling, whitening, dulling	no change	-
Perchloroethylene (tetrachloroethylene)							
99%	233	-	-	28	dulling, softening of surface	no change	-
99%	хт	-	-	1	swelling, slight chemical attack	no change	-
99%	XT-R	-	-	1	pronounced swelling + chemical attack	no change	-
Shellsol T							
	233	-	+	28	no change	no change	0
	ХТ	-	+	28	no change	no change	0
	XT-R	-	0	28	slight haze	no change	-
Turpentine substitute							
	233	-	+	28	no change	no change	0
	ХТ	-	+	28	no change	no change	0
	XT-R	-	-	7	swelling, whitening	no change	-
Turpentine oil DAB 7							
	233	-	+	28	no change	no change	0
	ХТ	-	+	28	no change	no change	0
	XT-R	-	-	7	swelling, whitening	no change	-
Carbon tetrachloride							
99%	233	-	-	1	swelling, slight chemical attack	no change	-
99%	хт	-	-	1	pronounced chemical attack	no change	-
99%	XT-R	-	-	1	partial dissolution	no change	-
Diesel fuel DIN 51601							
	233	-	+	28	no change	no change	0
	хт	-	+	28	no change	no change	0
	XT-R	-	0	28	colour change to	no change	-
					transparent brown	-	



FAM test fuel							
DIN 51604 A		_		<u> </u>			
	233	-	-	1	pronounced swelling, tackiness	no change	-
	ХТ	-	-	1	chemical attack, swelling, whitening	slight dulling, slight chemical attack	-
	XT-R	-	-	1	pronounced swelling, chemical attack	haze, chemical attack, swelling	-
FAM test fuel DIN 51604 B						j	
	233	-	-	1	chemical attack, swelling	slight haze	-
	хт	-	-	1	chemical attack, swelling	haze, chemical attack, swelling	-
	XT-R	-	-	1	chemical attack, swelling, whitening	haze, chemical attack, swelling	-
FAM test fuel DIN 51604 C							
	233	-	-	1	chemical attack, swelling	no change	-
	хт	-	-	1	chemical attack, swelling	haze, whitening, chemical attack	-
	XT-R	-	-	1	chemical attack, swelling, softening	haze, whitening, chemical attack	-
Fuel No. 1 DIN 53521							
	233	-	+	28	no change	no change	0
	XT	-	+	28	no change	no change	0
	XT-R	-	0	28	slight haze	no change	-
Fuel No. 2 DIN 53521							
	233	-	+	28	no change	no change	0
	XT	-	-	28	slight swelling	no change	-
Petrol, regular (unleaded)	XT-R	-	-	1	swelling, whitening	no change	-
(unieaueu)	233	1-	1.	28	swelling, yellowing	no change	-
	XT	-	-	7	swelling, dulling, softening	no change	-
	XT-R	-	-	1	swelling, colour change to brown, dulling	whitening of surface, dulling	-
Petrol, regular (leaded)							
	233	-	-	28	colour change to light brown	no change	-
	хт	-	-	28	swelling, colour change to light brown	no change	-
	XT-R	-	-	1	pronounced swelling, softening, colour change to brown	whitening of surface, dulling	-



Petrol, supergrade (unleaded)							
	233	-	-	28	swelling, yellowing	no change	-
	хт	-	-	7	swelling, dulling, softening	no change	-
	XT-R	-	-	1	swelling, colour change to brown, dulling	whitening of surface, dulling	-
Petrol, supergrade (leaded)							
	233	-	-	7	swelling, softening, yellowing	no change	-
	ХТ	-	-	1	swelling, dulling, softening	no change	-
	XT-R	-	-	1	very pronounced swelling, whitening	whitening of surface, dulling	-
Petroleum							
	233	-	+	28	no change	no change	0
	XT	-	+	28	no change	no change	0
	XT-R	-	0	28	haze, slight yellowing	no change	-

Acids, organic and inorganic

Chemical Conc	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term	OE
Citric acid				+	1	1031	
	000	1.					+
10%	233	+	+	28	no change	no change	· ·
10%	XT	+	+	28	no change	no change	+
10%	XT-R	+	0	28	specimens hazy, whitening	no change	0
38%	233	+	+	28	no change	no change	+
38%	XT	+	+	28	no change	no change	+
38%	XT-R	+	0	28	slight haze	no change	0
Formic acid							
5%	233		+	28	no change	no change	
5%	XT		+	28	no change	no change	
5%	XT-R		0	28	slight haze	no change	
Acetic acid							1
100%	233	-	-	1	specimens dissolved	no change	-
100%	хт	-	-	1	specimens dissolved	slight chemical attack	-
100%	XT-R	-	-	1	specimens dissolved	pronounced chemical attack, whitening	-
5%	233	+	+	28	no change	no change	+
5%	XT	+	+	28	no change	no change	+
5%	XT-R	+	0	28	specimens hazy, whitening	no change	0
Hydrofluoric acid							
40%	233	-	-	1	swelling, softening, whitening	slight swelling	-
40%	ХТ	-	-	1	swelling, softening, whitening	very slight dulling, swelling	-
40%	XT-R	-	-	1	swelling, softening, whitening	slight dulling, slight swelling	-



Lactic acid		_		+	· .	L .	_
20%	233	-	+	28	no change	no change	0
20%	XT	-	+	28	no change	no change	0
20%	XT-R	-	0	28	haze, whitening	no change	-
90%	233	-	-	7	pronounced swelling, whitening, softening	no change	-
90%	хт	-	-	1	pronounced chemical attack, whitening	no change	-
90%	XT-R	-	-	1	pronounced chemical attack, whitening	no change	-
Oxalic acid							
8,7%	233	+	+	28	no change	no change	+
8,7%	XT	+	+	28	no change	no change	+
8,7%	XT-R	+	0	28	haze, whitening	no change	0
Phosphoric acid					· · · •		
10%	XT	+	+	28	no change	no change	+
10%	233	+	+	28	no change	no change	+
10%	XT-R	+	0	28	haze, whitening	no change	0
50%	ХТ	-	+	28	no change	no change	0
50%	233	-	+	28	no change	no change	0
50%	XT-R	+	+	28	no change	no change	+
85%	233	-	-	1	pronounced swelling	no change	-
85%	хт	-	-	1	pronounced swelling, chemical attack	no change	-
85%	XT-R	-	-	1	pronounced swelling, chemical attack	no change	-
Nitric acid				+			
10%	233	+	+	28	no change	no change	+
10%	ХТ	+	+	28	no change	no change	+
10%	XT-R	+	0	28	yellowing, haze	no change	0
40%	233	-	+	28	no change	no change	0
40%	ХТ	-	+	28	no change	no change	0
40%	XT-R	-	-	28	colour change to opaque grey	no change	-
65%	233	•	-	1	very pronounced swelling, softening	dulling, whitening, swelling	-
65%	хт	-	-	1	very pronounced swelling, softening	dulling, whitening, swelling	-
65%	XT-R	-	-	1	very pronounced swelling, softening	dulling, whitening, swelling	-
Hydrochloric acid			_				
10%	233	+	+	28	no change	no change	+
10%	ХТ	+	+	28	no change	no change	+
10%	XT-R	+	o	28	whitening, haze	no change	0
32%	233	+	+	28	no change	no change	+
32%	XT	+	+	28	no change	no change	+
32%	XT-R	+	0	28	color change to grey, slight haze	no change	0



Sulphuric acid							
3%	233	+	+	28	no change	no change	+
3%	XT	+	+	28	no change	no change	+
3%	XT-R	+	0	28	whitening, haze	no change	0
30%	233	+	+	28	no change	no change	+
30%	XT	+	+	28	no change	no change	+
30%	XT-R	+	0	1	slight haze	no change	0
98%	233	-	-	1	pronounced swelling, whitening	dulling, whitening, swelling	-
98%	хт	-	-	1	pronounced swelling	dulling, whitening, swelling	-
98%	XT-R	-	-	1	pronounced swelling, reddening	dulling, whitening, swelling	-
Sulphamic acid (amidosulphonic acid)							
18%	233	+	+	28	no change	no change	+
18%	ХТ	+	+	28	no change	no change	+
18%	XT-R	+	0	28	haze, whitening	no change	0
Tartaric acid							
50%	233	+	+	28	no change	no change	+
50%	XT	+	+	28	no change	no change	+
50%	XT-R	+	0	28	haze, whitening	no change	0
Oleic acid							
99%	233	-	+	28	no change	no change	0
99%	XT	-	+	28	no change	no change	0
99%	XT-R	-	o	28	slight haze, dulling	no change	-

Alkalis

Chemical	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term	OE
Conc						test	
Ammonia solution							
10%	233	+	+	28	no change	no change	+
10%	XT	+	+	28	no change	no change	+
10%	XT-R	+	0	28	whitening (haze)	no change	0
25%	233	+	+	28	no change	no change	+
25%	XT	+	+	28	no change	no change	+
25%	XT-R	+	0	28	whitening	no change	0
Caustic soda solution							
1%	233	+	+	28	no change	no change	+
1%	XT	+	+	28	no change	no change	+
1%	XT-R	+	0	28	haze, whitening	no change	0
10%	233	+	+	28	no change	no change	+
10%	XT	+	+	28	no change	no change	+
10%	XT-R	+	+	28	no change	no change	+
30%	233	+	+	28	no change	no change	+
30%	XT	+	+	28	no change	no change	+
30%	XT-R	+	+	28	no change	no change	+



Salts	organic	and	inorganic	(saturated	solutions)
ouno,	organio	unu	morgame	Journarea	301410113/

Chemical Conc	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term test	OE
Aluminium chloride		1	1	1			1
42%	233	+	+	28	no change	no change	+
42%	ХТ	+	+	28	no change	no change	+
42%	XT-R	+	o	28	slight haze	no change	0
Ferric sulphate			_				
21%	233	+	+	28	no change	no change	+
21%	XT	+	+	28	no change	no change	+
21%	XT-R	+	0	28	haze, whitening	no change	0
Ferric chloride		-	-		The of the official states of the official st		Ť
48%	233	+	0	28	color change to light brown	no change	0
48%	хт	+	0	28	color change to light brown	no change	0
48%	XT-R	+	0	28	yellowing, haze, dulling	no change	0
Aluminium potassium sulphate							
5%	233	+	+	28	no change	no change	+
5%	XT	+	+	28	no change	no change	+
5%	XT-R	+	0	28	haze, whitening	no change	0
Potassium carbonate				1			ſ
50%	233	+	+	28	no change	no change	+
50%	XT	+	+	28	no change	no change	+
50%	XT-R	+	+	28	no change	no change	+
Potassium chloride			1				1
25%	233	+	+	28	no change	no change	+
25%	XT	+	+	28	no change	no change	+
25%	XT-R	+	+	28	no change	no change	+
Potassium nitrate	1	1	1	1	_		1
24%	233	+	+	28	no change	no change	+
24%	XT	+	+	28	no change	no change	+
24%	XT-R	+	0	28	haze, whitening	no change	0
Potassium		+	Ť	+			Ť
permanganate			1				1
6%	233	+	+	28	dulling, surface	no change	+
v /u	200	Ι.	1.	1	turning brown	Ino ondrige	1
6%	хт	+	+	28	dulling, surface	no change	+
0.70		1.	1	20	turning brown	Ino change	1
6%	XT-R	+	+	28	dulling, surface	no change	+
- ,.		`	1	1-0	turning black		1
Potassium sulphate			1	1			1
10%	233	+	+	28	no change	no change	+
10%	XT	+	+	28	no change	no change	+
10%	XT-R	+	0	28	slight haze	no change	0
Copper sulphate	n	1	1-	+			Ť
17%	233	+	+	28	no change	no change	+
17%	XT	+	+	28	no change	no change	+
17%	XT-R	+	0	28	haze, whitening	no change	0
Magnesium sulphate		+	Ť				Ť
21%	233	+	+	28	no change	no change	+
21%	XT	+	+	28	no change	no change	+
21%	XT-R	+	+	28	slight haze	no change	
Sodium acetate	R	+	-	20	anynii naze		0
	222	+	1_	20	no change	no oboneo	┟┰╴
32% 32%	233	+	+	28		no change	+
	XT	+	+	28	no change	no change	+
32%	XT-R	+	+	28	no change	no change	+

Sodium carbonate (soda ash)							
2%	233	+	+	28	no change	no change	+
2%	XT	+	+	28	no change	no change	+
2%	XT-R	+	0	28	specimens hazy, whitening	no change	0
20%	233	+	+	28	no change	no change	+
20%	XT	+	+	28	no change	no change	+
20%	XT-R	+	0	28	specimens hazy	no change	0
Sodium chloride			-				
(common salt)							
10%	233	+	+	28	no change	no change	+
10%	ХТ	+	+	28	no change	no change	+
10%	XT-R	+	0	28	haze, whitening	no change	0
Sodium phosphate							
20%	233	+	+	28	no change	no change	+
20%	ХТ	+	+	28	no change	no change	+
20%	XT-R	+	0	28	slight haze	no change	0
Sodium dihydrogen phosphate							
50%	233	+	+	28	no change	no change	+
50%	ХТ	+	+	28	no change	no change	+
50%	XT-R	+	0	28	very slight haze	no change	0
Disodium hydrogen phosphate							
8,5%	233	+	+	28	no change	no change	+
8,5%	ХТ	+	+	28	no change	no change	+
8,5%	XT-R	+	0	28	haze, whitening	no change	0
Sodium hydrogen sulphate							
40%	233	+	+	28	no change	no change	+
40%	ХТ	+	+	28	no change	no change	+
40%	XT-R	+	0	28	haze, whitening	no change	0
Sodium nitrate							
45%	233	+	+	28	no change	no change	+
45%	ХТ	+	+	28	no change	no change	+
45%	XT-R	+	0	28	slight haze	no change	0
Sodium sulphate (Glauber's salt)							
25%	233	+	+	28	no change	no change	+
25%	ХТ	+	+	28	no change	no change	+
25%	XT-R	+	0	28	haze, whitening	no change	0
Sodium chlorate							
49%	233	+	+	28	no change	no change	+
49%	хт	+	+	28	no change	no change	+
49%	XT-R	+	0	28	haze, whitening	no change	0
Sodium thiosulphate		1		<u> </u>			_
41%	233	+	+	28	no change	no change	+
41%	ХТ	+	+	28	no change	no change	+
41%	XT-R	+	+	28	no change	no change	+
Zinc chloride		<u> </u>			<u> </u>	<u> </u>	
50%	233	0	+	28	no change	no change	<u> </u>
50%	хт	0	+	28	no change	no change	0
50%	XT-R	+	0	28	haze, whitening	no change	0





Zinc sulphate							
35%	233	+	+	28	no change	no change	+
35%	ХТ	+	+	28	no change	no change	+
35%	XT-R	+	0	28	haze, whitening	no change	0
Urea							
51%	233	+	+	28	no change	no change	+
51%	ХТ	+	+	28	no change	no change	+
51%	XT-R	+	+	28	no change	no change	+
Hydroquinone							
6,7%	233	-	0	28	color change to transparent brown	no change	-
6,7%	хт	-	-	28	color change to opaque reddish brown	no change	-
6,7%	XT-R	+	-	28	color change to transparent brown	no change	0

Inorganic compounds

Chemical	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term	OE
Conc						test	
Hydrazine							
15%	233	+	+	28	no change	no change	+
15%	XT	+	+	28	no change	no change	+
15%	XT-R	+	+	28	no change	no change	+
Hydrogen peroxide							
(hydrogen dioxide, Perhydrol)							
3%	233	+	+	28	no change	no change	+
3%	ХТ	+	+	28	no change	no change	+
3%	XT-R	+	0	28	haze, whitening	no change	0
30%	233	+	+	28	no change	no change	+
30%	XT	+	+	28	no change	no change	+
30%	XT-R	+	0	28	haze, whitening	no change	0
Sodium hypochlorite							
12%	233	+	+	28	no change	no change	+
12%	XT	+	+	28	no change	no change	+
12%	XT-R	+	0	28	haze, whitening	no change	0
Water,							
demineralised							
	233	+	+	28	no change	no change	+
	ХТ	+	+	28	no change	no change	+
	XT-R	+	+	28	no change	no change	+

Organic compounds

Chemical Conc	Mat	RC	СВ	EZ	Evaluation of CR	CR, short-term test	OE
Dibutyl phthalate							
99%	233	-	-	28	chemical attack	no change	-
99%	XT	-	-	28	chemical attack	no change	-
99%	XT-R	-	-	1	swelling, chemical attack, whitening	no change	-



Diisobutyl phthalate							
97%	233	-2	+	28	no change	no change	
97%	XT		-	28	chemical attack	no change	
97%	XT-R	ĵ.		28	pronounced chemical attack, haze, crazing	no change	-
Paraffin, liquid		6	0		1		
100%	233	+	+	28	no change	no change	+
100%	XT	+	+	28	no change	no change	+
100%	XT-R	+	+	28	no change	no change	+
Di(2-ethylhexyl) sebacate (dioctyl sebacate)							
	233	-	+	28	no change	no change	0
	XT	-	+	28	no change	no change	0
	XT-R	-	+	28	no change	no change	0
Triorthocresyl- phosphate					- <u>-</u> -		
	233		+	28	no change	no change	0
	XT	-	-	28	no change	no change	-
	XT-R	-	2	7	chemical attack, dulling	no change	-
Rizinusöl							
98-90-000-00-00-00-00-00-00-00-00-00-00-00	233	-	+	28	no change	no change	+
	XT	-	+	28	no change	no change	+
	XT-R	•	+	28	no change	no change	0
Sojabohnenöl	E.	12	4		11 EA		
	233	-	+	28	no change	no change	0
	XT	-	+	28	no change	no change	0
	XT-R		+	28	no change	no change	0
Triethanolamin		1	3			and the second second	
98%	233	+	+	28	no change	no change	+
98%	XT	-	+	28	no change	no change	0
98%	XT-R	+	+	28	no change	no change	+

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12 Glossary

Comparative pressure measurement

During the sublimation, i.e. when the concentration of water vapour molecules is rather high in the atmosphere, the value that is provided by the gas-type dependent vacuum sensor of the "Pirani" type (e.g. Thyracont VSP 62/63) deviates from the value that is provided by a capacitive vacuum sensor (e.g. MKS 722B). When the proportion of water vapour molecules decreases towards the end of the main drying phase, the two sensors fall increasingly in line with one another.

The comparative pressure measurement is often used as a criterion for the automatic switching from the main drying phase to the final drying phase as well as for identifying the end of the process.

Desorption

Desorption (from Latin de-sorbere, sorbere = sup up, suck in) describes a phenomenon whereby molecules are released from the surface of a solid. In order to be able to desorb, the particle must have, or be provided with, a sufficient amount of energy in order to overcome the binding energy.

Eutectic point

The eutectic point is the point at which a homogenous mixture (e.g. a eutectic alloy) passes directly from the liquid to the solid phase without the formation of a crystal mixture that consists of different phases.

Pressure increase test

The pressure increase test can only be carried out with \rightarrow *double-chamber method.* During the pressure increase test, the intermediate valve prevents the flow of steam from the drying chamber to the ice condenser so that the water vapour of the \rightarrow *sublimation* cannot flow off. The result is a more or less distinct pressure increase that is measured in the product chamber. When the product has been completely dried, the vacuum does not decrease at all or only to a slight extent.

The pressure increase test is often used as a criterion for the automatic switching from the main drying phase to the final drying phase as well as for identifying the end of the process.

Single-chamber method

At the single-chamber method, the freezing as well as the subsequent drying of the product are both performed in the ice condenser chamber. The sample is frozen as a result of the low temperature of the ice condenser

 $(-55^{\circ}C \text{ in the case of one-stage systems or }-85^{\circ}C \text{ in the case of two-stage systems})$. The inside of the chamber can be cooled to approximately $-20^{\circ}C$ or $-40^{\circ}C$. The moderate supply of the frozen sample with energy, which is necessary during the main drying phase, is ensured by heatable shelves on which the product is placed.

12 Glossary



Double-chamber method

Drying on shelves outside the ice condenser chamber is referred to as a double-chamber system. The advantage compared to the \rightarrow single-chamber method is the considerably higher product capacity. In addition, the product chamber can be isolated from the ice condenser chamber by an intermediate valve in order to perform a so-called \rightarrow pressure increase test for determining the end of the drying process. In freeze-dryers without an active shelf cooling, the samples need to be pre-frozen externally, e.g. in a deep-freeze or freezer cabinet. After the transfer of the product into the freeze-dryer, the actual \rightarrow sublimation is started.

Reference designator

During the service life of industrial systems, a standardised reference designation system is required for the planning, design, realisation, maintenance, and disassembly stages in order to be able at all times to identify every single component within the system in an unambiguous manner. The reference designators) are affixed to the components and entered into the technical documentation (e.g. circuit diagrams).

Safety pressure

Since the vacuum has a dominating influence on the product temperature, Martin Christ Gefriertrocknungsanlagen GmbH has integrated a so-called safety pressure feature into the freeze-dryers in order to ensure the protection of the product. If the pressure inside the drying chamber increases too strongly so that it exceeds the safety limit, the energy supply of the shelves will be interrupted and the sublimation process slows down. This prevents the product from melting.

The safety pressure value that is entered should correspond to a temperature value that is 5°C below the melting point of the product on the vapour pressure curve above ice.

Sublimation

Sublimation (from Latin "sublimis" = high up in the air, raised), is a thermodynamic process of the direct transition of a substance from the solid phase to the gas phase.

Wireless Shelf Technology

With the Wireless Shelf Technology (WST), Martin Christ Gefriertrocknungsanlagen GmbH has developed a system for laboratory freezedryers that works without cable connections at the electrically heated shelves and other equipment parts. Instead, a connection plate with two electrical contacts is placed between the ice condenser chamber and the external drying chamber. This plate is the central plate for the connection of the accessories. Every electronic accessory is equipped with a separate module. These modules enable the combination of various components.



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