



INSTRUCTION MANUAL

ScanVac Freeze Dryers

All CoolSafe models Basic & Pro

INSTRUCTION MANUAL-CoolSafe Freeze Dryers

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Important user information

Please read this entire manual to fully understand the safe and effective use of this product.

In case you have any comments about this manual we will appreciate receiving them at the address below.

Warranty and Liability

LaboGene Aps. guarantees that the product delivered has been thoroughly tested to ensure that it meets its published specifications. The warranty included in the conditions of delivery is valid only if the product has been installed and used in accordance with the instructions supplied by LaboGene Aps..

LaboGene Aps. shall in no event be liable for incidental or consequential damages, including without limitation, loss of profits, loss of income, loss of business opportunities, loss of use, and other related exposures, caused by incorrect or in appropriate use of the product.

Symbols used in this manual

	WARNING Used in case of danger of a serious accident or when documentation needs to be consulted.
	NOTE Used to direct attention to a special item.

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Enclosure: Declaration of conformity

1. Introduction

The CoolSafe series of Freeze Dryers are small, compact and easy to use Freeze Dryer.

Using the wide range of accessories including a selection of chambers and manifolds, the CoolSafe's can be employed for a large variety of different applications.

Freeze-drying is often used for sample preparations and for preservation and storage of biological and pharmaceuticals materials. Freeze-drying is also ideally suitable for substances that are heat sensitive.

If you are not familiar with the freeze-drying process, please read Section 7, which outlines the freeze-drying process.

2. Safety precautions

2.1. General information

Please read this entire manual to fully understand the safe and effective use of this product.

Should you have any comments about this manual we will be pleased to receive them.

**NOTE**

If this equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

2.2. Safety

In case of overload, an automatic safety device stops the freeze dryer.

All CoolSafe models are electrically protected with an over current circuit breaker.

The safety system is designed according to Safety requirements for electric equipment, control and laboratory use (EN 61010-1), which assumes that two separate faults do not occur simultaneously.



NOTE LaboGene ApS. recommends using safety glasses for personal protection when connecting flasks or other equipment to the freeze-dryer – due to the risk of implosion.

3. Installation

3.1. Installation of the CoolSafe

- Make sure that the mains supply is according to the specifications on the type label at the rear panel.
- It is important that free air circulation is available at the air inlet and outlet grills. If the CoolSafe is

- positioned against a wall, there must be at least 100 mm free space at the rear of the unit.
- The unit is intended to be located on a tabletop or on the lower shelf of a trolley.

3.2. Installation of vacuum pump

A vacuum pump (two-stages with 2-5 m³/hour capacity) must be connected to the CoolSafe in order to allow cryo pumping.

LaboGene recommends the VacuuBrand RZ2.5 rotary vane pump for which a variety of protection accessories against aggressive materials are available. If the pump outlet/exhaust is not discharged via a fume hood, an oil mist filter should always be connected. See accessories section for recommended rotary vane pump and oil mist filter.

- Check that the mains supply for the pump is according to the specifications on the type label.
- Connect the vacuum hose to the suction side of the pump.
- Connect the pump to the vacuum connection pipe on the rear panel of the CoolSafe.
- Check the oil level on the pump and fill it if necessary. The oil level sight glass should be 1/2 - 3/4 full.
- LaboGene recommends that when the pump is running the gas ballast valve should be open.

3.3. Installation of accessories

The CoolSafe is a basic unit and must be equipped with the necessary accessories in order to run a specific freeze-drying process. Select from the wide range of accessories listed in the accessories section.

Make sure to mount the correct manifold, chamber and shelf arrangements and always check that all connections are tight.

4. Functional description

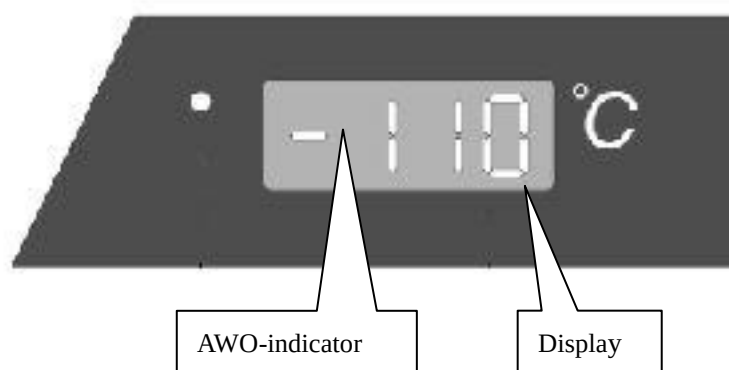
For reference, see the overview in section 6.

4.1. Mains

The main On/Off switch is on the front panel and mains cable connector is located on the rear of the unit.

4.2. Control panel CoolSafe Basic

CoolSafe Basic has the following indicators:



Display

A four-digit display shows condenser temperature in the range from ambient temperature down below -110 °C or -55°C, depending on the CoolSafe model.

Alarm-Wait-Ok (AWO) indicator

A multicolor LED shows one of the following states

State	LED Color	Description
Alarm*	Red Flashing	Condenser temperature rising above -20 °C or sensor malfunction. See Alarms and Error section.
Wait	Yellow Flashing	Condenser temperature between -20 °C and -70 °C.
Ok	Green	Condenser temperature below -70 °C / -90 °C initially.
*Alarm state is both visual and acoustic.		

4.3. Control panel CoolSafe Pro –SL12a02

SL12a02 is an electronic control system for ScanVac type CoolSafe Pro. User input and output is performed via a keyboard and display unit, the SL01a03.

The operator can control the system in a simple menu-structured way as described in the following text. Due to the many different ways to configurations the unit, the following paragraphs is only examples.

The keyboard unit consists of a keyboard with the keys 0-9, arrow UP and arrow RIGHT, and a display with 2x16 characters.

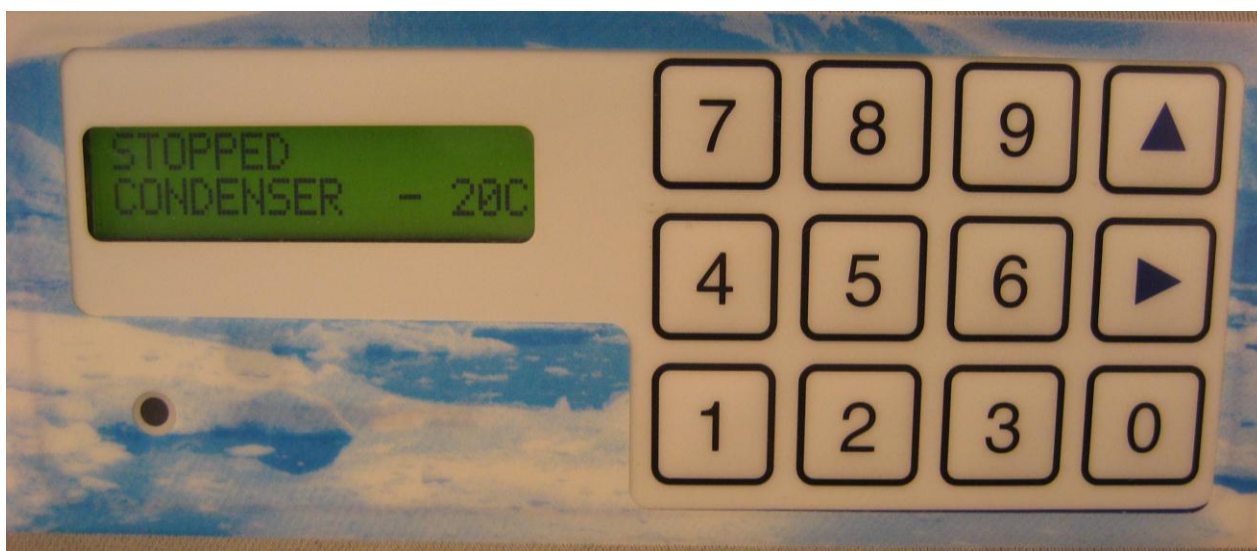
Generally, the RIGHT-key leads to the next menu and the UP-key is for selecting between different possibilities or is used to reset numbers or change their signs.

When in a menu, the red LED is on, except when the user is trying to input an out of range value, forcing the LED to blink. The LED is off when in NORMAL mode (=when not in a menu), except when an internal or external error is present, in which case the LED blinks.

NORMAL display:

In accordance with the selected modes (see later):

The display will show a description of the actual process on the first line, and, for example, a temperature related to this on the second line. Eventually browse between the possible variables related to the actual process with the UP-key. The browser is reset to its start position when pressing one of the numerical keys.

**MAIN menu:**

From the main display, key in RIGHT and SELECT MODE between MANUAL, AUTOMATIC, DE-ICE, RELEASE and STOP (note that not these entire mode is present on all models) pressing the UP key the appropriate number of times. When the desired menu is shown press RIGHT, and continue as described for each mode in the following paragraphs.

**MANUAL submenu:**

Select between COOLING STOP and COOLING START with the UP-key and proceed with the RIGHT-key.

A number of other menus may follow in accordance with the actual model.

Leaving the menu (common routine for all modes):

When the last menu is reached, select with the UP-key between END MENU WITH LEAVE (no changes will be made), END MENU WITH SAVE (any possible selections will be saved for later use) and END MENU WITH SAVE AND START (all possible selections will be saved and activated, and all other modes will be automatically cancelled). Finish then menus with the RIGHT-key.

**AUTOMATIC submenu:**

Input PREFREEZE, TIME=XX:XX.

Input the max. pressure, PRESSURE, MAX=XXXX.XXX hpa.

In accordance with the configuration:

Input the first shelf-interval, STEP 1, TIME=XX:XX in hours and minutes.

Input the related STEP 1, SHELFTEMP.=XX in degrees C.

Input the related STEP 1, PRESS.=XXXX.XX hpa (the selected value should be $\leq$ 90% off the earlier selected max-pressure).

These 3 steps are repeated a number of times.

Leave the menu as described under MANUAL submenu.

RELEASE submenu:

Leave the menu as described under MANUAL submenu.

DE-ICE submenu:

Input the DE-ICE, TIME =XX:XX in hour and minutes.

Leave the menu as described under MANUAL submenu.

STOP submenu:

Leave the menu as described under MANUAL submenu.

4.4. Valves and connections

- CoolSafe must be used with the top lid of transparent acrylic.
- Select the manifolds or chambers to be used from ScanVac freeze drying catalogue



- The drain valve is located at the front, and is used for draining the ice condenser after de-icing.



5. Operation

The freeze drying process is a balance between the heat absorbed by the sample to vaporize the ice, and the heat removed by the condenser to convert the water vapor into ice.

It is important to remember following factors when trying to obtain efficient utilization of the freeze dryer system.

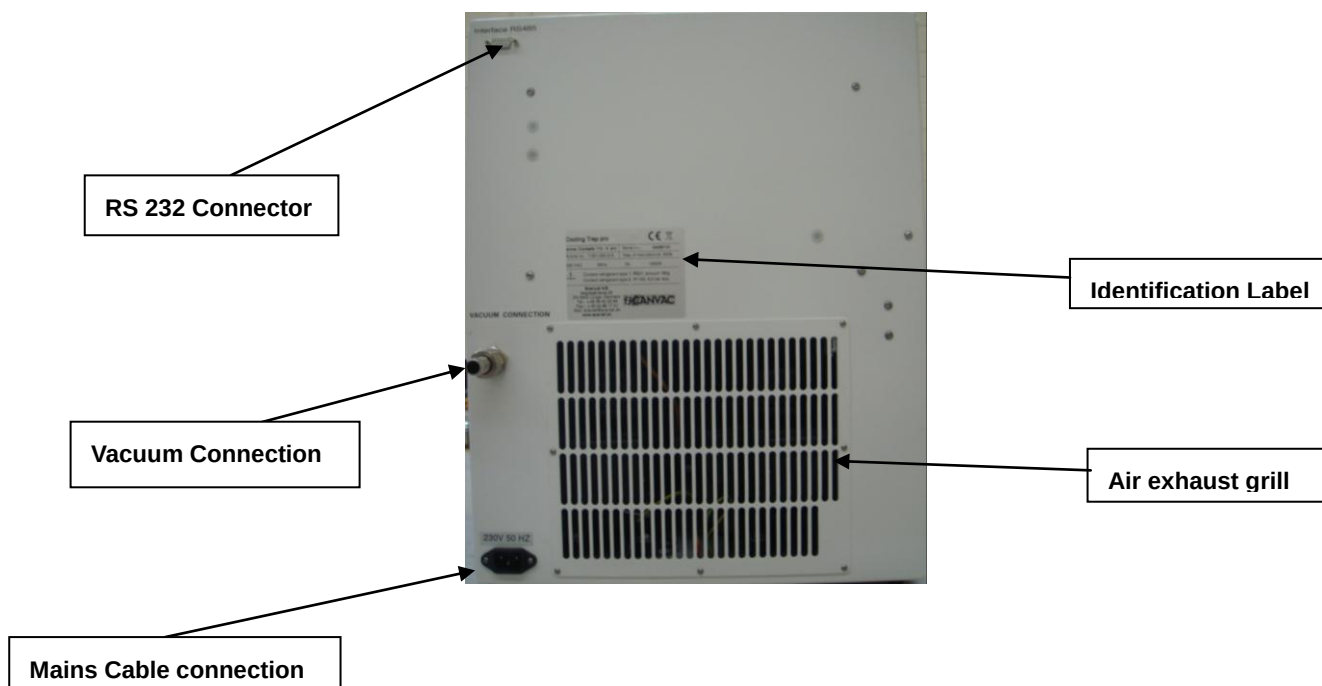
The efficiency of the freeze drying process depends upon:

- The surface area and the thickness of the sample.
- The temperature of the condenser and the vacuum obtained.
- The eutectic point (melting temperature) and the concentration of the sample solution.

5.1. Overview



Rear view:



5.2. Preparations

Check that the CoolSafe ice condenser is free from ice and water, and that all valves i.e. the drain valve is closed.. Check that there is no water in the drain valve.



NOTE

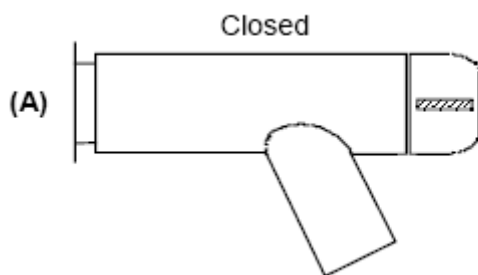
Do not use any tools to close valves, as they will easily be damaged.

- Check that the gas ballast valve on the vacuum pump is open at all times. Check the oil level in the pump and the oil mist filter. The filter must be operated with maximum 10-mm oil level (preferably empty).
- Switch on the CoolSafe and the vacuum pump. The CoolSafe will enter the WAIT-STATE indicated by a flashing yellow LED, and the cooling process starts. Just after switch on, the display shows the actual software version and then all display segments are switched on for a test the of display. After a few seconds, the temperature measurement is stabilized and the actual condenser temperature will be displayed.
- The vacuum pump should now be allowed to run for at least 30 minutes before opening the (external) vacuum valve. This period of time also allows the condenser to reach operational temperatures (below -90 °C or -55°C) indicated by the GREEN LED, which signals that the CoolSafe is ready and OK for freeze-drying to commence..
- The product has to be in frozen state using auxiliary equipment, i.e. freezer, liquid nitrogen etc or use the condenser as a freezer. To avoid product melting within the time it takes to move the product to the freeze dryer and establish a pressure below the triple point, the temperature of the product should be brought down to as low a temperature as possible.

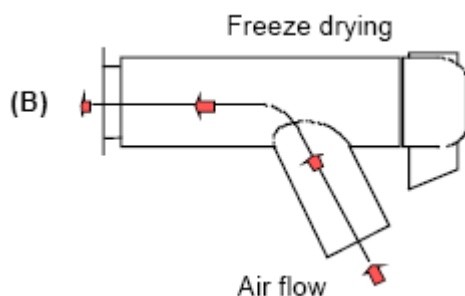
5.3. Freeze-drying using manifolds



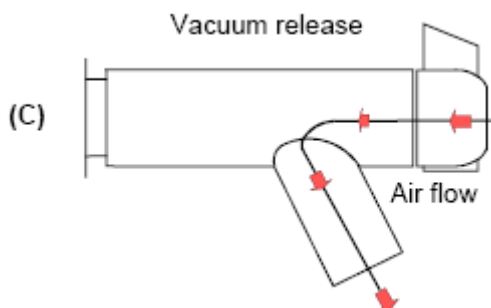
1. Check that all rubber valves are closed (A) or in vacuum-release position (C), and that the flange and lid are tight.



2. Start the application of the vacuum by opening the vacuum valve and check that the AWO-Indicator stays green indicating OK for freeze-drying.
3. Connect a flask to an appropriate cone on the rubber valve. Turn the rubber valve to position (B). The AWO-Indicator should always be green prior to connecting the next flask. The freeze-drying process is now in progress.



4. To stop the freeze-drying process, turn the rubber valve handle 180° to position (C) to equalize the pressure in the flask, and remove the flask from the cone.



5. Close the vacuum valve after removal of the last flask.
6. To allow the system to equalize the vacuum in condenser, use the rubber valve.

5.4. Freeze-drying using chambers



1. Make sure that the (external) vacuum valve is closed.
2. Mount the chamber and shelf arrangement on the CoolSafe. Check that all connections are tight and the chamber is closed. If heated shelves are to be used, refer to the controller manual.
3. Open the chamber; place the frozen material on the shelves in the chamber. Close the chamber and the drain valve.

	NOTE Make sure that both drain valve and release valve are closed.
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4. Open the (external) vacuum valve. The freeze-drying process is now in progress.
5. To stop the freeze-drying process, close the (external) vacuum valve and equalize the pressure by opening the release valve at the lid.
6. Remove the material from the shelves.

5.5. De-ice

The freeze dryer may be de-iced by:

- Leaving the ice condenser open at room temperature.
- Filling the ice condenser with hot water leaving the drain valve open until the ice has melted or take the ice block out after 1 minute.

	NOTE Do not fill the ice condenser with too much water
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5.6. Stopping the vacuum pump

	NOTE Keeping the pump at operation temperature and with open gas ballast valve minimises the risk for harmful condensation of vapours in the pump. Do not stop the vacuum pump during the freeze drying process!
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Close the (external) vacuum valve and let the pump run for a minimum of 45 minutes after the freeze-drying has ended. This is to avoid any possible condensates remaining in the pump oil.

If the freeze dryer is used regularly during the week, it is recommended that the vacuum pump runs constantly with gas ballast valve open and the vacuum valve closed.

6. Alarms and errors

In case of alarms or errors, an alarm/error code will be displayed.

E_1: Error indicating that the temperature sensor has a short circuit.

E_2: Alarm indicating that the temperature during freeze-drying is above 20 °C.

E_3: Error indicating that the temperature sensor is disconnected.

7. Maintenance

7.1. CoolSafe

The air-cooled condenser must be cleaned minimum twice a year. Use a vacuum cleaner and remove any dust in the cooling grill on the front.

Rubber valves, black O-rings and valves on the manifold and O-rings on chambers should be greased with a ultra thin layer of vacuum grease and wiped off with a soft cloth every 3-4 months.

The rubber must appear shiny, smooth and without cracks. All silicone gaskets needs only dust removal (do not use any vacuum grease). If necessary clean with alcohol wipes.

7.2. Vacuum pump

See the vacuum pump instruction manual for maintenance according to supplier's recommendation.

Maintenance for a vacuum pump in general will be to:

- Check the oil volume and colour condition regularly. The oil should be changed every 400-500 working hours, more often if the pump has been exposed to aggressive vapors. Keep a Log book of the usage.
- Drain the oil mist filter if the oil level is more than 10 mm.
- Drain, clean and eventually change the filter insert if smoke comes from the oil mist filter.

7.3. Cleaning

	NOTE When using strong materials such as strong acids or bases, radioactive substances and volatile organic solvents it must be handled carefully and promptly cleaned up if spilled.
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If a sample is spilled in the concentrator chamber, it must be cleaned up immediately.

8. The Freeze-drying process

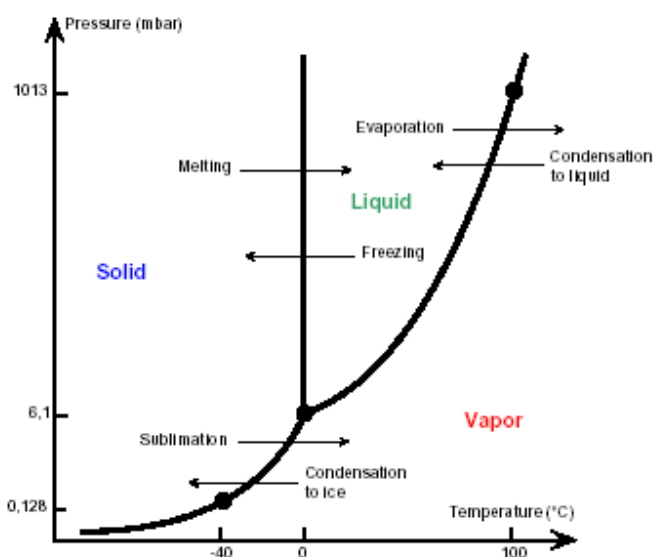
To understand the freeze-drying process, this section describes the theory for the process in short.

For more detailed information about freeze-drying please consult available literature e.g. “ An introduction to freeze drying by James M. Flink & Henning Knudsen, ISBN 87-872-0084-8.

All material in free form will exist in one of three states: Vapor, liquid or solid.

As shown in the diagram for water, the physical state is dependent on the surrounding pressure and the temperature. The point where all three phases of water co-exist is called the triple point.

Freeze-drying is basically a change in state and properties of the material that is to be removed. In principle the process is very simple, but in reality it is can be very complex as the product can consist of many components, which interact with each other.



Moving from one state to another, requires the supply or removal of energy to or from the product. This energy flux will have an impact on the structure of the material being dried.

Freeze-drying is achieved using equipment that controls temperature and pressure in the environment of the product and is a 3-step process.

- In the first step, the product is frozen solid so that the water present in the material is converted to ice (pre-freeze).
- In the second step, the ice formed in pre-freezing is removed from the product by direct conversion from solid to vapor. This process is called sublimation. In order to start removing moisture in the sublimation process, the pressure surrounding the material has to be brought down to a value below that of the triple point, whilst keeping the temperature of the product below its freezing point. The condenser surface temperature must also be down to a temperature lower than the temperature of the material handled. When the product is heated it will theoretically place itself somewhere on the

transition line between the solid and vapor states. The heating will initiate sublimation of the moisture from the product to the surrounding environment. The vapor will move to the condenser surface because of the lower vapor pressure in the condenser.

- In the third step, water that was strongly bound to the solids of the sample (called sorbed water) is converted to vapor and removed from the product, this process being referred to as desorption. Removing moisture in the desorption phase or secondary drying phase, is done at a higher product temperature and at the lowest possible pressure.

8.1 Pressure of Aqueous Vapor

PRESSURE OF AQUEOUS VAPOR

Pressure of aqueous vapor over
ice in hPa for temperatures
from -98 to 0°C

Vapor Pressure of Ice

Temp	Press	Temp	Press	Temp	Press
0.0	6.103	-12.4	2.097	-24.8	0.647
-0.2	6.003	-12.6	2.060	-25.0	0.634
-0.4	5.906	-12.8	2.023	-25.2	0.622
-0.6	5.810	-13.0	1.986	-25.4	0.609
-0.8	5.714	-13.2	1.951	-25.6	0.597
-1.0	5.620	-13.4	1.915	-25.8	0.585
-1.2	5.527	-13.6	1.880	-26.0	0.573
-1.4	5.436	-13.8	1.847	-26.2	0.562
-1.6	5.347	-14.0	1.814	-26.4	0.551
-1.8	5.259	-14.2	1.780	-26.6	0.540
-2.0	5.171	-14.4	1.748	-26.8	0.529
-2.2	5.086	-14.6	1.716	-27.0	0.518
-2.4	5.002	-14.8	1.684	-27.2	0.508
-2.6	4.919	-15.0	1.654	-27.4	0.499
-2.8	4.838	-15.2	1.624	-27.6	0.488
-3.0	4.755	-15.4	1.594	-27.8	0.479
-3.2	4.677	-15.6	1.566	-28.0	0.468
-3.4	4.599	-15.8	1.536	-28.2	0.459
-3.6	4.522	-16.0	1.508	-28.4	0.449
-3.8	4.446	-16.2	1.480	-28.6	0.439
-4.0	4.371	-16.4	1.454	-28.8	0.431
-4.2	4.298	-16.6	1.426	-29.0	0.422
-4.4	4.226	-16.8	1.400	-29.2	0.414
-4.6	4.154	-17.0	1.374	-29.4	0.405
-4.8	4.085	-17.2	1.348	-29.6	0.397
-5.0	4.016	-17.4	1.323	-29.8	0.389
-5.2	3.948	-17.6	1.299	-30.0	0.381076
-5.4	3.881	-17.8	1.274	-32.0	0.308966
-5.6	3.814	-18.0	1.251	-34.0	0.249652
-5.8	3.749	-18.2	1.227	-36.0	0.200868
-6.0	3.685	-18.4	1.204	-38.0	0.161147
-6.2	3.622	-18.6	1.182	-40.0	0.129758
-6.4	3.561	-18.8	1.159	-42.0	0.102366
-6.6	3.500	-19.0	1.138	-44.0	0.081173
-6.8	3.440	-19.2	1.116	-46.0	0.064112
-7.0	3.381	-19.4	1.095	-48.0	0.050393
-7.2	3.322	-19.6	1.074	-50.0	0.039387
-7.4	3.265	-19.8	1.054	-52.0	0.030656
-7.6	3.209	-20.0	1.034	-54.0	0.023725
-7.8	3.154	-20.2	1.014	-56.0	0.018394
-8.0	3.100	-20.4	0.995	-58.0	0.014128
-8.2	3.045	-20.6	0.977	-60.0	0.010769
-8.4	2.993	-20.8	0.958	-62.0	0.008184
-8.6	2.941	-21.0	0.939	-64.0	0.006184
-8.8	2.889	-21.2	0.921	-66.0	0.004651
-9.0	2.840	-21.4	0.903	-68.0	0.003478
-9.2	2.789	-21.6	0.886	-70.0	0.002585
-9.4	2.741	-21.8	0.869	-72.0	0.001906
-9.6	2.693	-22.0	0.853	-74.0	0.001399
-9.8	2.645	-22.2	0.835	-76.0	0.001026
-10.0	2.599	-22.4	0.819	-78.0	0.000746
-10.2	2.553	-22.6	0.803	-80.0	0.000533
-10.4	2.509	-22.8	0.789	-82.0	0.000386
-10.6	2.464	-23.0	0.773	-84.0	0.000266
-10.8	2.421	-23.2	0.758	-86.0	0.000186
-11.0	2.379	-23.4	0.743	-88.0	0.000133
-11.2	2.336	-23.6	0.729	-90.0	0.000093
-11.4	2.295	-23.8	0.714	-92.0	0.000064
-11.6	2.253	-24.0	0.701	-94.0	0.000044
-11.8	2.213	-24.2	0.686	-96.0	0.000029
-12.0	2.175	-24.4	0.673	-98.0	0.000020
-12.2	2.135	-24.6	0.659		

Dok.: 00029740

9. Technical Specifications & Accessories

See attached catalogue on homepage www.labogene.com/scanvac

Declaration of Conformity

We declare under our responsibility, that the following product

CoolSafe, Models
CoolSafe 110-4, CoolSafe 110-4 Pro, CoolSafe 55-4, CoolSafe 55-4 Pro, CoolSafe 55-9,
CoolSafe 55-9 Pro, CoolSafe 100-9 Pro, CoolSafe 55-15 Pro, CoolSafe 95-15 Pro
CoolSafe 90-80 Superior

to which this declaration relates is in conformity with the following standard(s)
directives or other normative document(s):

EN ISO 12100-1: 2003 Safety of machinery
(basic concepts – General principles)

EN ISO 12100-2: 2003 Safety of machinery
(Technical principles and specifications)

EN 60204-1: 2006 Safety of machinery
(Electrical equipment of machines – General requirements)

EN 61010-1: 2001 Safety requirements for electrical equipment for measurement,
control and laboratory use. General requirements

EN 61000-6-3: 2007, EN 61000-6-1: 2002
Electromagnetic compatibility
(Generic immunity/emission standard)

EN ISO 14121-1: 2007 Safety of machinery
Risk assessment - Part 1: Principles
EN ISO 11201 – Acoustics – Noise emitted by machinery and equipment

following the provisions of:

EU Medical Devices Directive 93/42/EEC

Directive 2006/42/CE Machinery safety

Directive 2006/95/EEC, per August 2007
(Electrical equipment designed for use within certain voltage limits)

Directive 2004/108/EEC EMC Electromagnetic compatibility

Lynge, June 2011

A handwritten signature in blue ink, appearing to read "Finn Kokholm".

Finn Kokholm
Managing Director



LaboGene ApS, Industrivej 6-8, Vassingerød, 3540 Lynge, Denmark