

# THE PROCESS

# PRE-FREEZING, PRIMARY DRYING & SECONDARY DRYING

# THE BASIC PRINCIPLE:

Freeze drying is a process whereby a product is dried by removing the water under low temperature and pressure.

# THE TECHNICAL EXPLANATION:

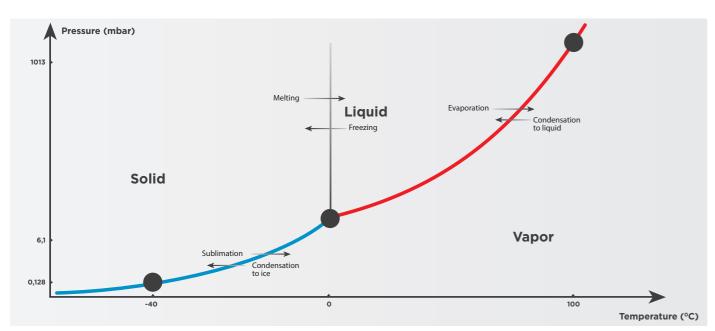
Freeze drying involves the removal of water or other solvents from a given product by a process called sublimation. This occurs when the ice of a frozen product converts directly to the gaseous state without passing through the liquid phase. This enables the preparation of a stable product that is easy to use and store at ambient temperatures.

A low pressure environment is a pre-requisite to allow this process to take place. In order to start the removal of the water, the pressure inside the freeze dryer must be below the "triple point value" for the product, whilst also maintaining the temperature of the sample below its freezing point.

# FIRST STAGE - PRE-FREEZING:

The sample is frozen, which means the water in the product is converted to ice, thereby the phase has changed from liquid to solid.

Slow pre-freezing will produce larger ice crystals, which are easier to freeze dry, whilst fast pre-freezing results in smaller crystals.



# SECOND STAGE - PRIMARY DRYING:

In the second stage the sublimation process starts. The ice formed in the pre-freeze step is removed from the sample by the direct transition of the "solid" ice to a vapour without passing through a liquid phase. The resultant vapour is collected in the condenser, which has a lower temperature and pressure than the product. The vapour is thus converted back to ice on the condenser surface.

The "energy" required for this process to occur is provided by a gentle heating of the sample, which will start the sublimation process and eventually the sample will dry.

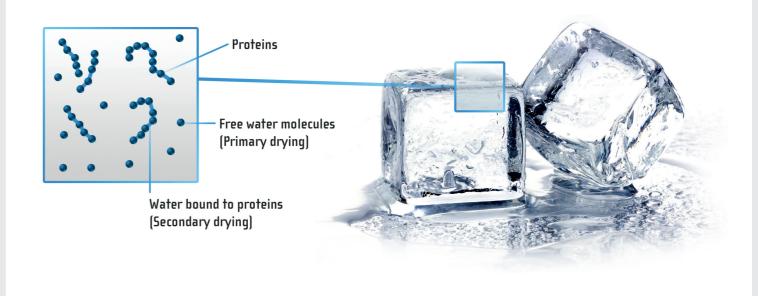
If too much energy (heat) is applied to the sample during this stage the condenser may not be able to condense the volume of vapours fast enough, the ice condenser temperature will subsequently rise along with its vapor pressure, thus increasing the risk of the sample melting.

# THIRD STAGE - SECONDARY DRYING:

Finally, any residual water present, which is strongly bound to the molecules of the sample, is converted to vapour and removed from the sample.

This water has invariably a vapour pressure lower than that of water in its "free" form.

Removal of the water in this final stage will be performed at higher product temperatures, consequently, any biological activity of the sample will not be impaired or affected. This usually involves increasing the temperature and lowering the pressure to provide enough energy to break down the molecular bonding. A process called desorption.



# CHOOSING

# **YOUR FREEZE DRYER**

# HOW TO CALCULATE THE SIZE OF THE FREEZE DRYER

When the size of the freeze dryer is to be determined several questions have to be addressed, for example:

- What condenser size (volume) is required?
- What condenser temperature is required?
- What is the maximum number of samples to be freeze dried at one time?
- What is the total volume of the samples?
- What is the size of the container/tray you want to use for the samples?
- What is the sample volume in each tube/vial?

If "bulk drying" on trays or shallow containers, the sample depth should ideally be less than 1,5 cm. When all these questions have been answered the size and configuration of the freeze dryer can then be determined.





# WHAT SIZE OF CONDENSER IS REQUIRED?

The size of a freeze dryer is rated in size by the capacity of the condenser to hold ice that is produced in a 24 hour drying cycle (i.e. water from the sample)

As a minimum, the condenser capacity must be adequate to handle the total amount of vapor from the sample volumes in a single batch before de-icing is required.

The larger the condenser's surface area, the more efficiently vapor is condensed to ice and, consequently, the thinner the ice-layer formed.



# WHAT CONDENSER TEMPERATURE IS NEEDED?

The choice of condenser temperature is important! If the temperature is too high, then a correct freeze drying process cannot take place.

In the table to the right are the freezing points/collapsing temperatures of some commonly used solvents and known products. These are given for guidance purposes and to assist in deciding the condenser temperature for your application.



If the condenser temperature is near to the freezing point of the sample, then the freeze drying cycle times are extended.

The product could possibly start to melt, thus giving very poor recovery rates and enhancing the possibility of damaging the vacuum pump, eventually polluting the laboratory environment. This phenomenon is known as a "collapse" of the sample.

The driving force in freeze drying is expressed as the temperature/pressure differential between the product and the condenser. In conclusion, the lower the condenser temperature, the better! For example, for aqueous samples a -55C condenser temperature is the preferred choice.

However, a lower temperature condenser will provide faster freeze drying, avoiding melting and give better results, with the additional benefit of protecting the vacuum pump and the environment.

Collapsing temperatures of different products				
Products	Temperatures			
Apple Juice	-42°C			
Citrate Buffer	-40°C			
Coffee Extract	-20°C			
Dextran	-9°C			
Fruktose	-48°C			
Gelatin	-8°C			
Glucose	-40°C			
Inisitol	-27°C			
Lactose	-32°C			
Lemon Juice	-36°C			
Methocel	-9°C			
MSG	-50°C			
Orange Juice	-24°C			
Ovalbumim	-10°C			
Phosphate Buffer	-8°C			
PVP	-23°C			
Sorbitol	-45°C			
Sucrose	-32°C			
Solutions containing ethanol etc.	-60°C110°C			
Trichlorethylene	-86,4°C			



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# The process of selecting the correct freeze dryer for your application is divided into 8 steps:

- Step 1: Temperature, volume, voltage, surface of the condenser and the preferred freeze dryer version - Basic, Pro, Touch or Touch Superior.
- Step 2: This will guide you to the particular model based on your requirements.
- Step 3-8: Select the type and size of the chamber, type and size of a manifold and any accessories you need for your freeze drying application.



# **Temperature:**

You can choose between -55°C, -95°C, -100°C and -110°C condenser temperatures. Please refer to the information on the previous pages with regards to which temperature is optimal for your freeze drying requirements.



# Volume:

Based on the chosen temperature you have different condenser volumes to select from.

	-55°C	-95°C	-100°C	-110°C
4 litre	X			X
9 litre	X		X	
15 litre	X	X		
80 litre	X	X		

# CONFIGURATION

through the configurator and be guided Check the complete process!

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# BASED ON YOUR CHOICE OF TEMPERATURE AND VOLUME SELECT THE MODEL MOST SUITED FOR YOUR REQUIREMENTS:

	Basic	Pro	Touch	Superior Touch XS/XL	Superior Touch
-55°C and 4L	X	X	X		
-55°C and 9L	X	X	X		
-55°C and 15L		X	X		
-55°C and 80L					X
-95°C and 15L		X	X	X	
-95°C and 80L					X
-100°C and 9L		X	X	X	
-110°C and 4L	X	X	X		

# **BASIC**

Digital read-out of temperature. Included are hose and flange for connections to the vacuum pump and the vacuum solenoid valve, which is controlled from the front of the freeze dryer.

# **PRO**

Digital read-out of temperature and pressure. Included are hose and flange for connections to the vacuum pump and the vacuum solenoid valve, which is controlled from the front of the freeze dryer.

# **TOUCH**

For Manual and Automatic operation, with optimum control and regulation of temperature, pressure and time. Included are vacuum hose and flange connection to the pump.

# All operations are controlled from the touchscreen display, including:

• Vacuum solenoid valve.

- Electrical de-icing.
- Pressure regulation valve for primary drying.
- Software back-up.

Vacuum release valve.

# **SUPERIOR TOUCH**

For manual and automatic operation, with optimum control and regulation of temperature, pressure and time, with the additional facility of pre-freezing. Included are vacuum hose and flange connection to the pump.

# All operations are controlled from the touchscreen display, including:

Vacuum solenoid valve.

- Electrical de-icing.
- Pressure regulation valve for primary drying.
- Software back-up.

• Vacuum release valve.

The XS models are inclusive of an electrical heated three-shelf rack for pre-freezing. For the XL models, you can choose between a version inclusive of an electrical heated three-shelf rack and a version inclusive of an electrical heated five-shelf rack. See page 18 for the distances between the shelves.









CoolSafe Superior Touch XL/XS



**CoolSafe Superior Touch** 

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# **VOLTAGE:**

You have the possibility of choosing between 115V/60Hz and 230V/50-60Hz. For an 80 litre condenser the only option is 3x400V/50-60Hz.

# Surface of the condenser

Some of our freeze dryers can be supplied with teflon coated condensers for use when for freeze drying materials that may contain aggressive acids. Please contact us for further information and advice.

# STEP 2

# The freeze dryer version

Based on your selection of temperature, volume, model, voltage and surface of the condenser you will have specified the exact model, which best suits your exact requirements and needs.







# STEP 3

**TOP LIDS** -Select the lid required for your application

# For CoolSafe Basic, Pro and Touch



# Acrylic lid (ACpl)

Acrylic plate on the top of the CoolSafe including vacuum release.

This facilitates all non-heated chambers (CCS200 and 300) and manifold M4 basic.



CCS 300 lid with hole for M4 basic manifold Extra top plate for CCS 300 with drilled hole for adaption

of M4 Basic.

# STEP 4

**MANIFOLDS** -Select the manifold required for your application

# For CoolSafe Basic, Pro and Touch



# Manifold 4 Basic

Manifold Basic with 4 rubber valves 3/4 inch for freeze drying in flasks. (Can be extended with the addition



# Manifold 4 Extension

Manifold extension including 4 rubber valves 3/4 inch for freeze drying in flasks.





# Complete M4 Manifold

- +Manifold 4 Extension
- + Acpl



# Drum manifold

Drum manifold made of stainless steel.

Included are 12 3/4 inch rubber valves.

The ACpl lid is not necessary to use this manifold.



# Ampoule manifold 16

Ampoule manifold made of stainless steel for 3/4 inch rubber valves with 16 x 6 mm pipes. Included is 1,5 meter



# **Additional information**

Please contact the LaboGene distributor in your specific country or visit our webpage www.labogene.com



# For CoolSafe Superior Touch 80 L



Manifold 5

Side manifold for CoolSafe Superior Touch 55°C/95°C with an 80 litre capacity with 5 quick seal valves 3/4 inch.



Manifold 14

Manifold for CoolSafe Superior Touch 55°C/95°C with an 80 litre capacity with 14 quick seal valves 3/4 inch for freeze drying in flasks.

# STEP 5

# **CHAMBERS**

# Non-heated chambers

- to be used with the Basic, Pro or Touch models.

Various sizes and formats are available which offer complete flexibility of use. These include options for stoppering, attachment of flasks, and racks with removable shelves.

# **Chamber CCS 200**



# Chamber CCS 200, ø200 mm

Polycarbonate chamber ø200 mm including 2 x ø180 mm adjustable stainless steel shelves and trays.

Distance between shelves: Min. 20 mm with 6 shelves. Max. 105 mm with 2 shelves. (Possibility to have up to 6 shelves and trays).



#### Rack 200

Extra rack for CCS 200 including 2 shelves and trays for pre-freezing.



Tray 200

Extra stainless steel tray ø180 mm x 18 mm for CCS 200 chamber.





# Shelf ø180 mm

Extra stainless steel shelf for CCS 200 chamber.

Note it only works as support for the tray.

# **Chamber CCS 300**



Chamber CCS 300, ø300 mm Acrylic chamber ø300 mm with 3 stainless steel shelves

ø250 mm and 3 trays with adjustable height.

Distance between shelves: Min. 28 mm with 12 shelves. Max. 328 mm with 2 shelves. (Possibility to have up to12 shelves and trays).



# Chamber, CCS 300 4V, ø300

Acrylic chamber ø300 mm with 3 stainless steel shelves ø250 mm, 3 trays with adjustable height and  $4 \times 3/4$ inch rubber valves.

Distance between shelves: Min. 28 mm with 12 shelves. Max. 328 mm with 2 shelves. (Possibility to have up to 12 shelves and trays).



# Chamber, CCS 300 8V, ø300

Acrylic chamber ø300 mm with 3 stainless steel shelves ø250 mm, 3 trays with adjustable height and 8 x 3/4 inch rubber valves.

Distance between shelves: Min. 28 mm with 12 shelves. Max. 328 mm with 2 shelves. (Possibility to have up to 12 shelves and trays).



# Chamber, CCS 300 with stoppering arrangement

Acrylic chamber ø300 mm with 3 shelves ø250 mm with mechanical stoppering arrangement.

Distance between shelves: Min 20 mm Max. 78 mm Total 0,15 m2 shelf area.





# Shelf ø250 mm

Extra stainless steel shelf for CCS 300 chamber.

Please note that the shelf only acts as support for the tray.



# Tray 300

Extra stainless steel tray ø250 mm x 21 mm for CCS 300 chamber.





Rack 300

Extra rack for CCS 300 including 3 shelves and trays for pre-freezing.



Rack micro-titre plate

Rack for 3 x 14 micro-titre or 3 x 7 micro-titre plates for CCS 300 chamber.

Art no.: 7001000387



# **Chamber CCS 500**



# Chamber, CCS 500, Ø500 mm Acrylic chamber Ø500 mm. The internal height is 480 mm.

Included are 3 stainless steel shelves and trays with adjustable height.

Distance between shelves: Min. 39 mm with 9 shelves. Max. 318 mm with 2 shelves. (Possibility to have up to 9 shelves and trays.)

Lid with 40 NV flange and NV 40 flex hose for connection to the CoolSafe are also included.

Art no.: 7001500085



# Tray 500

Extra stainless steel tray ø435 x 23 mm for CCS 500 chamber.

Art no : 7001500285



# Shelf ø440 mm

Extra stainless steel shelf for CCS 500 chamber.

Art no: 7001500185



#### **Heated chambers**

- to be used with the CoolSafe Touch and Superior Touch XL & XS

We offer a wide selection of chambers with electrical heated shelves that ensure uniform and faster drying, especially in the secondary drying stage. Giving increased recovery rates with controlled programmable energy input.

# Versatility with Adaptability!

# **Chamber CCS 300 E**



# Chamber, CCS 300 E, ø300 mm. heated

Acrylic chamber ø300 mm with 5 heated shelves ø250 mm.

Distance between shelves: Min. 38 mm with 5 shelves. Max. 89 mm with 3 shelves. (no. 2 and no. 4 shelves have been removed).

Included are the connection kit and product sensor.





# Chamber, CCS 300 8VE, ø300 mm, heated

Acrylic chamber ø300 mm with 5 heated shelves ø250 mm and 8 3/4 inch rubber valves.

Distance between shelves: Min. 38 mm with 5 shelves. Max. 89 mm with 3 shelves. (no. 2 and no. 4 shelves have been removed).

Included are the connection kit and product sensor.

#### Art no · 7001300184



# Tray 300

Extra stainless steel tray Ø250 mm x 21 mm for CCS 300 E chamber.

Art no.: 7001000285



# Chamber, CCS 300 4VE, ø300 mm. heated

Acrylic chamber ø300 mm with 5 heated shelves ø250 mm and 4 3/4 inch rubber valves.

Distance between shelves: Min. 38 mm with 5 shelves. Max. 89 mm with 3 shelves. (no. 2 and no. 4 shelves have been removed).

Included are the connection kit and product sensor.

#### Art no.: 7001300083



# Chamber, CCS 300 E Stop, heated

Acrylic chamber ø300 mm with 3 heated shelves ø250 mm and stoppering arrangement.

Distance between shelves: Min. 20 mm Max. 78 mm.

Included are the connection kit and product sensor.

# Art no.: 7001300197

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# Chamber CCS 500 E



# Chamber, CCS 500 E, ø500 mm. heated

Acrylic chamber ø500 mm. The internal height is 480 mm. Included are 2 electrical heated shelves ø435 mm with adjustable height.

The minimum distance is 65 mm with 5 electrical heated shelves.

Please note that the distance(s) increase when the shelf or shelves are removed.

Included are the connection kit and product sensor. Lid with 40 NV flange and NV 40 flex hose for connection to the CoolSafe are also included.



Tray 500 Extra stainless steel tray ø435 x 23 mm for CCS 500 E chamber.

# Racks for CoolSafe Superior Touch XS and XL

XS (9 L volume/1 kg. ice capacity) and XL (15 L volume/2 kg. ice capacity)

The standard model XS is supplied with an electrical heated three-shelf rack for pre-freezing. For model XL, you can either choose a version supplied with an electrical heated three-shelf rack or a version supplied with an electrical heated five-shelf rack.

This provides a fully automatic research freeze drying system that incorporates the highest performance characteristics and technical features to meet the demands of today's research, analytical and development laboratories.



### Superior Touch XS

Freeze dryer including rack with 3 heated shelves ø180 mm.

Distance between shelves is 34 mm.

Contact us for further information if teflon coating is needed.

Art no. for XS 230V: 700059 Art no. for XS 115V: 700060



# Superior Touch XL 3 Shelves

Freeze dryer including rack with 3 heated shelves ø180 mm.

Distance between shelves is 79 mm.

Contact us for further information if teflon coating is needed.

Art no. for XL 3S. 230V: 700061 Art no. for XL 3S. 115V: 700062



### Superior Touch XL 5 Shelves

Freeze dryer including rack with 5 heated shelves ø180 mm.

Distance between shelves is 34 mm.

Contact us for further information if teflon coating is needed.

Art no. for XL 5S. 115V: 703179

# Chambers for CoolSafe Superior Touch (80 L volume / 10 kg. ice capacity)

CoolSafe Superior Touch which has a condenser capacity of 80L can be configured with bulk rack or vial rack with heated shelves for pre-freezing. This provides a fully automatic research freeze drying system that incorporates the highest performance characteristics and technical features to meet the demands of today's research, analytical and development laboratories.



Vial rack Superior Touch

Vial rack including the stoppering arrangement pneumatic and 2 electrical heated shelves (250 x 400 mm) for stoppering of vials. It takes up to 5 shelves.

Distance between shelves: (without/with radiation shelf) 2 shelves mounted: 106/101

3 shelves mounted: 67/63 mm 4 shelves mounted: 47/45 mm 5 shelves mounted: 36 mm





Compressor up to 6 Bar, 230V/50-60 Hz

Compressor up to 6 Bar for the stoppering arrangement.

Can be purchased locally.





Shelf 2540 for Superior Touch

Extra electrical heated shelf 2540 (250 x 400 mm). Each shelf is 0,10 m2, and a maximum of 3 extra shelves can be added.



Tray 2540 for Superior Touch

Tray 2540 in stainless steel for operation in vials rack. (30 x 250 x400 mm).





Bulk Rack Superior Touch no. 1 Bulk rack including 2 electrical

heated shelves (300 x 500 mm). It accommodates up to 5 shelves.

Distance between shelves: Min. 35 mm with 5 shelves.



Tray 3050 for Superior Touch Bulk no. 1

Tray 3050 in stainless steel for bulk drying (30 x 300 x 500 mm).



Bulk rack Superior Touch no. 2

Bulk rack including 2 electrical heated shelves (300 x 500 mm), with a maximum of up to 8 shelves. Distance between shelves: Min. 18 mm with 8 shelves. Max. 200 mm with 2 shelves.

Art no.: 7001001897



Tray 3050 for Superior Touch for Bulk no. 2

Tray 3050 in stainless steel for bulk drying (15  $\times$  300  $\times$  500 mm).

Art no.: 7001001802



# Shelf 3050 for Superior Touch Bulk Extra electrical heated shelf 3050 (300 x 500 mm).

Each shelf is 0,15 m2. A maximum of 3 extra shelves can be added for Bulk no. 1 and 6 extra shelves for bulk no. 2.

Art no: 7001001801

# STEP 6

# **PUMPS**

Select the vacuum pump most suited for your freeze dryer/chamber combination.



# Vacuum pump, RZ 2.5

30 L/min and 2,5 m3/hour. The ultimate vacuum is 0,001 mBar with anti-blow back valve. Included is the oil mist filter – art no.: 7001500071.

Art no. for 230V/50Hz version: 7001500069 Art no. for 110V/60Hz version: 7001506069



# Vacuum pump, RZ 6

105 L/min and 6,0 m3/hour. The ultimate vacuum is 0,001 mBar with anti-blow back valve.
Included is the oil mist filter – art no.: 7001500071.

Art no. for 230V/50Hz version: 7001510070 Art no. for 110V/60Hz version: 7001516070



# Vacuum pump, RC 6 Chemistry hybrid pump Chemistry hybrid vacuum pump. 5.9/6.9 m3/hour.

The ultimate vacuum is 0,002 mBar. Included is the oil mist filter – art no.: 7001500071.

Art no. for 230V/50Hz version: 7001510071



# Vacuum pump, RZ 9

140 L/min and 8,6 m3/hour. The ultimate vacuum is 0,0002 mBar. Included is the oil mist filter – art no.: 7001201070.

Art no. for 230V/50Hz version: 7001201169

# STEP 7

# **ACCESSORIES**

Practical accessories for your consideration.



#### **SVM 85**

ScanVac Vacuum Meter, absolute pressure is 1200-0.001 mBar, with data-logging facility, NV 10 and flange. Including connection kit for SVM 85.

For CoolSafe Basic.

#### Art no.: 700100026



# Trolley, freeze drying

Compact trolley for one CoolSafe and Vacuum pump.

#### Art no: 7001100950



# Castors

Set of 4 pieces, with 2 which are lockable. For mounting on a freeze dryer. It can be mounted on the following versions: CoolSafe 55-4, CoolSafe 55-9 and CoolSafe 110-4.

Art no.: 7001000066



# Connection kit for 2 chambers

T-kit with 2 x  $\frac{3}{4}$  inch rubber valves for connection of 2 chambers to 1 vacuum pump.

#### Art no: 7001000077



# Gate valve

Valve enabling closure between CoolSafe and a ø500 chamber.

#### Art no.: 7001500879



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# STEP 8

# **FLASKS**

Choose between round bottom and normal freeze drying flasks.

# Freeze drying flasks

# Round bottom freeze drying flasks



#### Flacks 150 m

Freeze drying flasks/chamber 150 mL with rubber lid for connection to 3/4 inch rubber valves, ø52 x 60 mm.

#### Art no.: 7001200100

# Flasks 300 mL

Freeze drying flasks/chamber 300 mL with rubber lid for connection to 3/4 inch rubber valves,  $Ø52 \times 120$  mm.

#### Art no.: 700120010

# Flasks 600 mL

Freeze drying flasks/chamber 600 mL with rubber lid for connection to 3/4 inch rubber valves, Ø75 x 100 mm.

#### Art no: 7001200103

#### Flasks 1200 mL

Freeze drying flasks/chamber 1200 mL with rubber lid for connection to 3/4 inch rubber valves, Ø75 x 200 mm.

Art no.: 7001200103



# Round bottom flasks 250 mL

Round bottom 250 mL freeze drying flasks for cone 29/32

#### Art no.: 7001200105

#### Round bottom flasks 500 mL

Round bottom 500 mL freeze drying flasks for cone 29/32.

#### Art no.: 7001200106

# Round bottom flasks 1000 mL

Round bottom 1000 mL freeze drying flasks for cone 29/32.

#### Art no: 7001200107

# Round bottom flasks 2000 mL

Round bottom 2000 mL freeze drying flasks for cone 29/32.

Art no : 7001200108

# Cone connectors, that enables freeze drying flasks to connect directly to manifold valves.

Round bottom freeze drying flasks



# Cone 24/29

3/4 inch aluminium cone 24/29.

#### Art no.: 700100007

# Cone 29/32

3/4 inch aluminium cone 29/32.

### Art no.: 700100007

# Cone 34/36

3/4 inch aluminium cone 34/36.

Art no.: 700100007



# Spin freeze drive

Spin freeze drive for spin freezing of flasks for freeze drying.

#### Art no.: 700100037



Having completed the configuration procedure, you will have the freeze dryer tailored to your specific requirements.

# Why not try our online configurator on our webpage!

This will guide you through the complete process, and will also enable you to connect with our distributor in your particular country.

For additional information, please contact the LaboGene distributor in your specific country or visit our webpage www.labogene.com

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