

# USER'S MANUAL

## Laboratory Freeze Dryer CRYODOS



**TELSTAR**

CRYODOS\_I\_rev0105US

Telstar Industrial, S.L.  
Josep Tapiolas, 120  
08226 TERRASSA (Spain)  
Tel. +34 937 36 16 00  
Fax: +34 937 85 93 42  
telstar@etelstar.com  
www.etelstar.com

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## 1. GENERAL DETAILS

This Operation Manual is valid for laboratory Freeze dryer models:

***CRYODOS -50***

***CRYODOS -80***

### WARNING

- It is mandatory to read and fully understand this manual before using the unit.
- All safety instructions contained herein must be respected.
- Before carrying out any maintenance work on the equipment, it must be disconnected from the energy source.
- Use or application of the equipment that does not conform to the recommendations in this manual may cause danger and lead to the loss of the manufacturer's guarantee.

### MANUFACTURER:

**TELSTAR INDUSTRIAL, S.L.**  
Josep Tapiolas, 120  
08226 – TERRASSA (BARCELONA)

Telf. +34-93-736-16-00  
Fax. +34-93-785-93-42

Web: [www.etelstar.com](http://www.etelstar.com)  
E-mail: [telstar@etelstar.com](mailto:telstar@etelstar.com)

## 2. DETAILS OF THE FREEZE DRYER

### 2.1. AIM OF THIS MANUAL

The main aim of this manual is to give the user guidelines for the handling and operation of the **TELSTAR CRYODOS –50** and **CRYODOS –80**.

### 2.2. DESCRIPTION

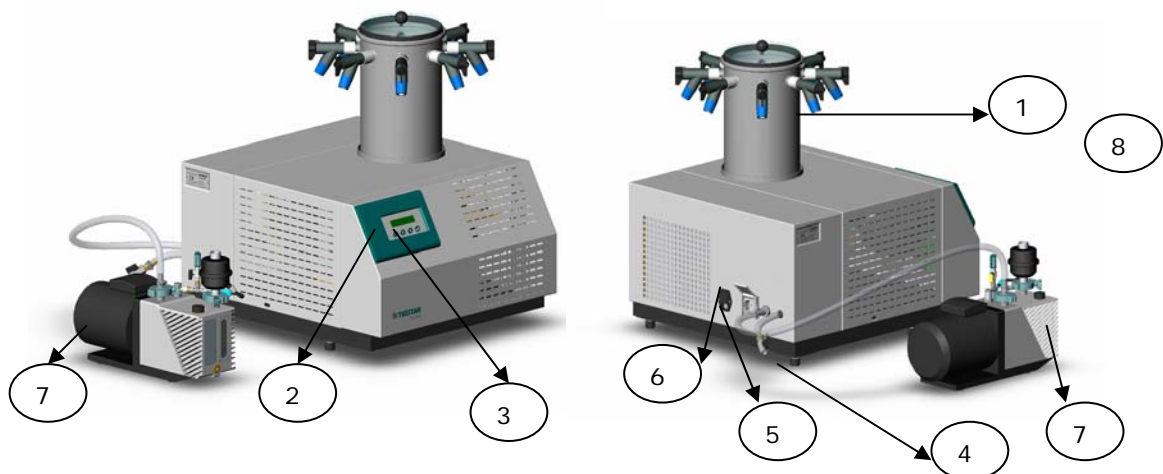
The CRYODOS freeze dryer in a compact desktop version. The basic unit comprises a metal cabinet, with a door at the back, which gives access to the inside for maintenance of all the components. The vacuum pump is delivered separately from the unit

#### Cryodos components

- One two-stage vacuum pump provided with Gas-Ballast. It holds inside a closing valve which, when the pump stops, cuts the pump off from the freeze dryer unit and keeps it under vacuum, while atmospheric pressure is re-established inside the pump, so that the oil cannot flow back. It also has an exhaust filter.
- One cooling system, cooling the condenser coil down to working temperature. The condensation surface is designed to a size that ensures condensation of the vapour given off by the product being freeze dried, preventing it from reaching the vacuum pump.
  - **CRYODOS –50**: one air condensing one-stage hermetic compressor.
  - **CRYODOS –80**: two air condensing one-stage hermetic compressors.

At the top, there is a stainless steel cylinder with 8 3/4" (18.7 mm) cocks and key. The cylinder has a top cover made of a transparent acrylic material to enable inspection of the condenser during the process. Each of the 8 cocks is equipped with its 3-way valve providing cutting off and re-establishing atmospheric pressure in each individual vial. These enable every ground vial mouths to be connected with the tapering adapter they hold. (Outside diameter, 29-32 mm – Inside diameter, 18 mm). Tubes and vials can be inserted directly when this adapter is removed.

On the front, there is the control panel composed of a LCD alphanumeric viewer showing the freeze-drying cycle menu. During the whole process, a screen with the condenser temperature and vacuum value will appear, inserting every 3 seconds another that shows the process time.



- |                             |                           |
|-----------------------------|---------------------------|
| 1.- Condenser with 8 cocks  | 5.- Electrical connection |
| 2.- LCD alphanumeric viewer | 6.- Main switch           |
| 3.- Pressing buttons panel  | 7.- Vacuum pump           |
| 4.- Draining pipe           |                           |

### **2.3. APPLICATION**

Using the various operative systems that make up the unit, and the possibility of connecting several accessories, this freeze-dryer can be used for freeze drying in flasks, vials and bottles or in bulk.

#### **WARNING**

- Under no circumstances may the unit be used with corrosive, inflammable or explosive gases.
- In the event of freeze drying product containing corrosive or explosive solvent, please contact the Technical Service, with a view to taking the specific preventive measurements for the product in question.

### **2.4. REQUIRED SERVICES**

Power supply: 230 V single-phase + earth, 50 Hz / 230 V single-phase + earth, 60 Hz.

Power capacity installed: 1 kW for the CRYODOS –50 and 1,5 kW for the CRYODOS –80.

Consumption (Amps): 5,5 A for the CRYODOS –50 and 8,5 A for the CRYODOS –80.

Maximum working room temperature: +30°C.

## 2.5. TECHNICAL FEATURES

VACUUM PUMP			CRYODOS –50 50 Hz / 60 Hz	CRYODOS –80 50 Hz / 60 Hz
Model			VARIAN DS 102	
Dimensions		LxWxH	430 x 132 x 212	
Weight		Kg.	22	
Suction flow		m³/h	5 / 6	
Motor velocity		r.p.m.	1500 / 1800	
Electric consumption		kW	0,45 / 0,55	
Oil	Load capacity	Litres	0,5	
		Type	AV-30	
ICE CONDENSER				
Condensing surface		m²	0,136	0,136
Final temperature		°C	-50	-80
Ice capacity		kg.	4 (1,5 kg/24 h)	4 (4 kg/24 h)
Condenser unit material			AISI – 316L	
REFRIGERATING UNIT				
Nr. of compressors			1	2
Hermetic type			Single-stage compressor	
Refrigerating power		W	450	900
Refrigerant free of CFCs			R-404a	R-404a / R-23
Assembly refrigeration			By air circulation	
UNIT				
Electrical connection		V/ph/hz	230/1/50 230/1/60	230/1/50 230/1/60
Power capacity installed (unloaded)		kW	1	1,5
Vacuum at the pump inlet		mbar	5 x 10 <sup>-3</sup>	5 x 10 <sup>-3</sup>
Process vacuum		mbar	4 x 10 <sup>-2</sup>	1 x 10 <sup>-3</sup>
Condenser at –45°C (CRYODOS -50)				
Condenser at –75°C (CRYODOS -80)				
With room temperature not higher than +21°C				
Dimensions	High	mm	700	700
	Width	mm	660	660
	Depth	mm	730	730
Vacuum gauge			DIGITAL	DIGITAL
PT100 Temperature probe			DIGITAL	DIGITAL
Weight		Kg.	60	90

### **3. *TRANSPORT***

The freeze-dryer is supplied as an individual package and in a suitable way to prevent it from being damaged, during transportation.

With the freeze-dryer is supplied the vacuum pump separately crated, including technical documentation and 1-litre bottle of oil. A tin of silicone oil is also supplied, for proper condenser seals maintenance.

When unpacking the freeze-dryer, check if the supply includes optional accessories, each of which would be separately packed to ensure suitable protection.

This manual is also included.

## 4. CAUTIONS BEFORE START-UP

For a correct start-up procedure, the following instructions must be followed step by step.

### 4.1. POSITIONING

The unit may be placed directly on a table if the surface is perfectly smooth and horizontal. The base unit must be firmly settled to prevent vibration.

### 4.2. ELECTRICAL CONNECTION

- Connect the electrical cable of the unit to the power supply.
- Check that the supply voltage is the correct one for the unit.
- The connecting cable is supplied with European plug.

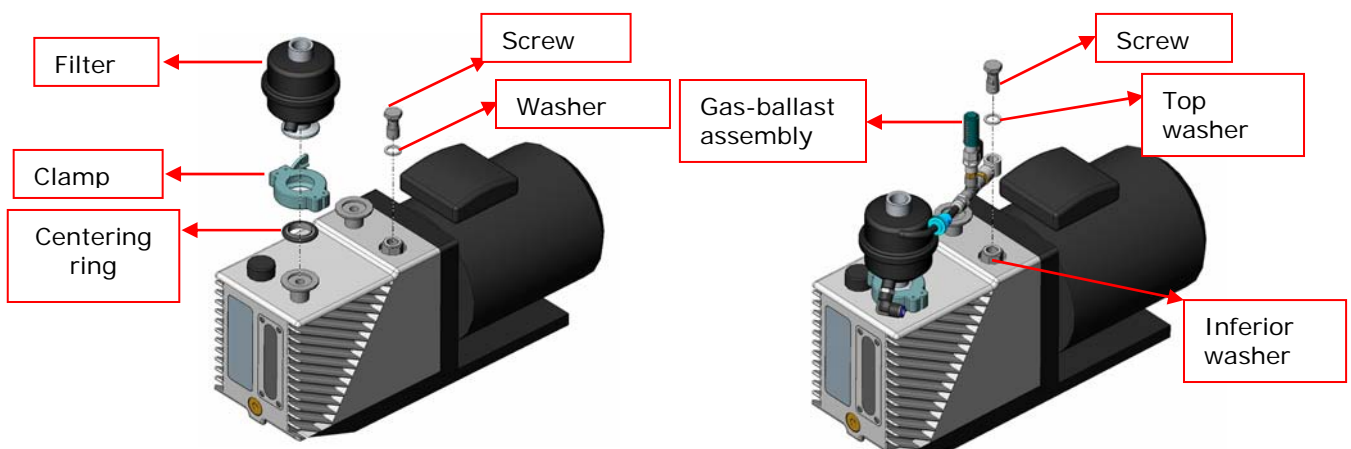
### WARNING

- Before switching the freeze dryer on, check that the supply voltage is the same as that mentioned on the specification plate.
- Whenever there is a power cut or the supply fails for a long period, stop the Freeze Dryer.
- Installation in the place where the Freeze-Dryer is connected must comply with current safety regulations according to each country.

### 4.3. VACUUM PUMP

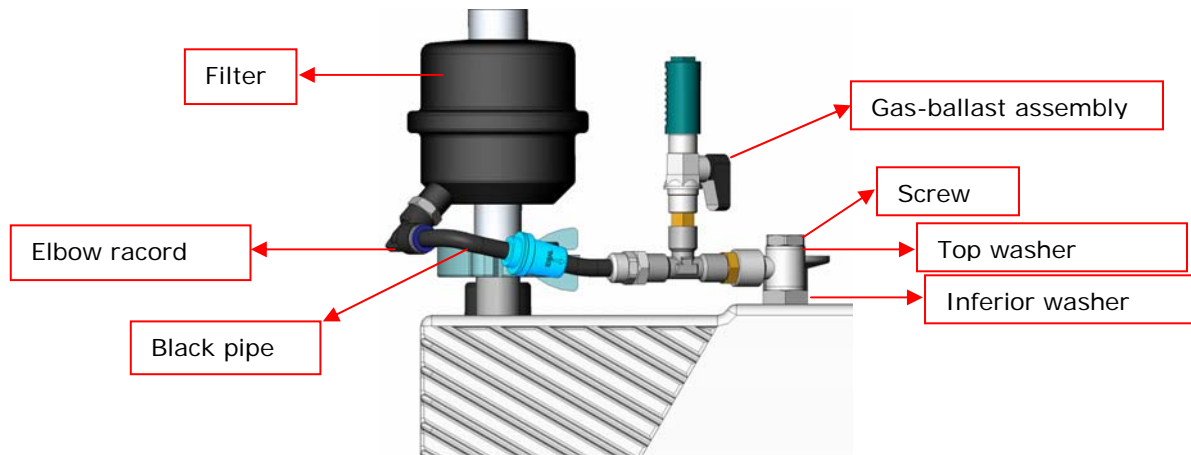
Steps to follow in order to assembly the filter and the gas-ballast. This operation must be carried out before the start-up of the vacuum pump.

- Connect the oil return filter to the vacuum pump by means of the centering ring and the clamp to the DN25 connection
- Take out the screw and a oil return connection washer.





3. Insert the gas-ballast assembly to the screw and thread to the oil return connection.



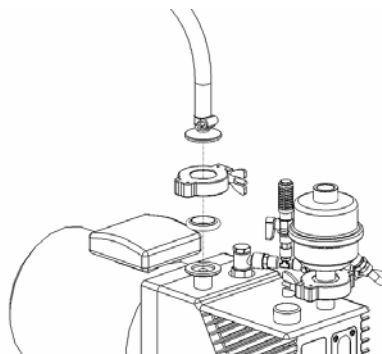
4. Connect the black pipe to the elbow racord placed in the connection filter base.

Pour new oil into the filling hole. Verify the level. Vacuum pump oil capacity: 0,5 litres (see paragraph 2.1.3. maintenance manual).

It is recommended to open  $\frac{1}{4}$  the gas ballast during the lyophilisation cycle in order to prevent product vapours not to sublime in the condenser. If this occurs, the vacuum pump oil can contaminate

The vacuum pump is equipped with an exhaust filter, which job is to retain oils vapours.

The electrical connection of the vacuum pump has to be connected to the base placed in the backside of the unit. Furthermore, the vacuum pump admission has to be connected to the base unit through the supplied pipe.



#### **4.4. ICE CONDENSER**

Before each operation, open the condenser water drain valve to ensure that the condensed ice from the previous operation has been totally removed. If any water remains in the condenser or drainpipe, it will freeze as a result of the vacuum, thus possibly compromising work conditions.

#### **4.5. REFRIGERATING UNIT**

In order to ensure that the refrigerating unit works properly (refrigeration via forced air circulation by means of a fan), all vents must be separated, minimum 20 cms. away from other equipment or the wall.

#### **4.6. LUBRICATION**

Slightly grease the airtight seals with the silicone grease. The contact surfaces should always be kept cleaned and greased. Avoid dirtiness accumulation.

#### **4.7. CONNECTION OF THE VACUUM PUMP DISCHARGE**

In the event that smoke from the vacuum pump is ducted outside, connect this tube to the vacuum unit discharge, avoiding the installed filter.

#### **4.8. LONG STORAGE / PERIODS OF NON-USE**

In the event that the freeze-dryer is placed in storage for a long time, the vacuum pump oil must be changed to ensure that there is no corrosive waste that could damage it. Disconnect the unit from the electricity supply. Clean the inside of the chamber, check that the condenser has been completely emptied and lubricate the chamber and the condenser's gaskets.

## 5. WORKING PRINCIPLE

### 5.1. FREEZING

Products must be frozen before they are freeze-dried. The layer of product should be as thin as possible (recommended inferior than 20 mm.). The length the freeze drying process depends essentially on this height and the solid composition of the product. Fully recommended are: even loading of the freeze dryer and equal heights in different containers of product to dry. For the same reason, a mixture of products to dry should be avoided.

Freezing temperature is proportionate to product solidifying point. Freezing speed influences the size of the crystals and, consequently, sublimation speed. Therefore, the most suitable freezing parameters for each product must be determined. It is advisable to freeze products at a temperature below  $-20^{\circ}\text{C}$  for a period of between 3 and 4 hours.

### 5.2. FREEZE DRYING

When the condenser reaches the programmed temperature and the vacuum pump is running, the bottles or flasks with the frozen product are now connected to the condenser cylinder valves. Open the valves so that the vacuum is produced in the container and product sublimation starts. As soon as all the flasks have been connected and valves opened, there should be a vacuum of less than 10 mbar in 4 minutes; otherwise, the pump will stop, appearing the alarm of the vacuum system. If this happens, check the valve-to-bottle connections for leaking. To continue with the process, press the ENTER key.

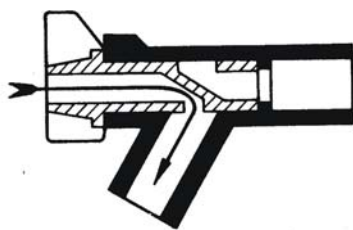
When wishing to connect the flask when the freeze drying process has begun, proceed as below:

1. Connect the new flask and open this valve.
2. Wait for a while until a vacuum is recovered in the unit.
3. Connect the next flask.
4. When all the flasks have been connected, let the equipment run until the product has been totally dried out.

If exterior heating has to be applied to the product while it is dried out to speed up the drying process, the heating source should be at the right distance to apply the radiation heat that the product requires. Heat from hot air directed directly onto the flasks may also be used.

At the beginning of the process, a layer of ice will form on the outside wall of the flask and this will gradually disappear as drying out continues. Total disappearance of product ice is indicated when the flask is at ambient temperature showing that the process has finished. When it has finished, the appropriate manifold cocks are closed and air enters in the flask or flasks, so they can be removed.

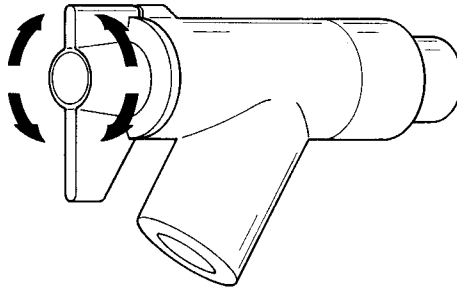
#### CONNECTING VALVE



Air inlet to flask



Vacuum to flask



The length of the main drying process during freeze drying, depends essentially on:

- The heating temperature that can be applied to product (outside the equipment).
- The maximum heating temperature accepted by the product.
- The final degree of humidity desired.
- The percentage of solid material in the product.
- The vacuum level obtained.
- The total amount of product to be dried each time.
- Condenser temperature.

### **5.3. AIR ENTERING DRYING CHAMBER AT END OF THE PROCESS**

Stop the process by pressing the ON/OFF key, which stops the pump and cooling unit and opens to let air in through some of the taps. Place a recipe at the end of the draining tube and remove the stopper. De-freezing can be sped up by applying hot air to the condenser coil. In any case, it is advisable to remove the cylinder with the valves to accelerate coil ice de-freezing.

Before starting a new process, ensure that the condenser is totally defrost, there are no remains of water in the drainpipe and that the valve is once again closed.

## **6. ACCESSORIES**

### **1- Stoppering chamber.** Ref. 7801

Glass cylindrical chamber (Ø 200 mm.) with 3 non thermostated shelves and manual vial closing device. Includes 3 trays (for vials and serum).

### **2- Chamber.** Ref. 8956

Glass cylindrical chamber (Ø 200 mm.) with 3 shelves and 3 trays (for vials, serum bottles and raw material).

### **3- 8 port manifold.** Ref. 70205

Including 8 rubber three way valves CN29/32 for flasks.

### **4- 40 port manifold.** Ref. 7917

Manifold with 40 ports for tubes of 13 mm. Ø. REMARK: To install this manifold is necessary to install the adapter plate for manifolds.

### **5- Adapter plate for manifold.** Ref. 7820

Adapter to place manifolds with connection DN-50.

## **7. OPERATING INSTRUCTIONS**

### **7.1. PREVIOUS OPERATIONS BEFORE START-UP**

- Check that all the water remains from de-freezing during the previous operation has been removed by opening the draining valve. Close again.
- Check that the condenser transparent cover is on.
- Check that the all the cocks of the chamber are turned off.

### **7.2. CONTROL PANEL**



### **7.3. INSTRUCTIONS FOR START-UP**

To start the equipment, proceed as follows:

Set the switch on placed in the back side, just above the electric inlet plug to position I.

The following screen will appear:

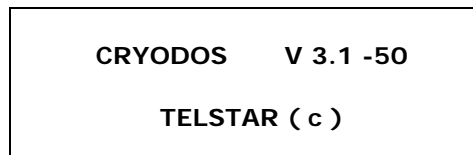


Fig. 1

After a few seconds the main screen will show:

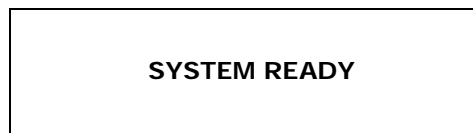


Fig. 2

Press ON/OFF key and the screen will show:

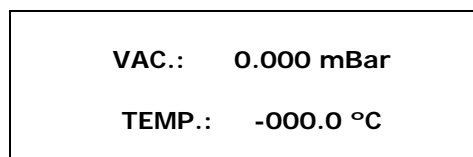


Fig. 3

The vacuum level inside the chamber and temperature in the condenser will appear.

At this point, the following process automatically begins:

- The cooling system starts (compressor 1).
- In the CRYODOS –80, compressor 2 starts 4 minutes after compressor 1 has already started.
- When the condenser reaches a programmed temperature (T), the vacuum pump automatically starts.

T=-30°C in the CRYODOS-50

T=-45°C in the CRYODOS-80

REMARK: These pump starting Temperature Points are set and cannot be modified by the user.

Every 3 seconds Fig. 3 will be merge with Fig. 4.

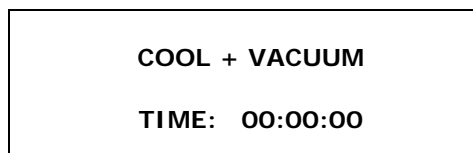


Fig. 4

#### 7.4. STOPPING THE FREEZE DRYER

Both models, when the freeze drying process ends, the equipment is stopped by pressing the ON/OFF key. Compressor 1 will continue running for 2 minutes to collect the system fluid refrigerant.

##### Accidental freeze dryer stopping.

If there is in the CRYODOS –50 an accidental stop, disconnection or power cut, the freeze dryer stops and the vacuum in the chamber is maintained. Once the current is re-established, the process continues working at the same conditions before the stop.

If it happens in the CRYODOS –80 and once the current is re-established, compressor 2 will run after 4 minutes of the start-up.

#### 7.5. REMARKS ON EQUIPMENT OPERATION

**Auxiliary screens.** There are four auxiliary time counters viewing screens, for: compressor 1, compressor 2 (CRYODOS –80 only), vacuum pump and language selection. To accede to the auxiliary screens, simply press the UP/DOPWN keys one after the other being the freeze dryer at rest, before pressing the ON/OFF key, so that "SYSTEM READY" is shown.

If there is a delay of more than 5 seconds in pressing the UP/DOWN keys on any of these screens, the program automatically goes back to the main screen.

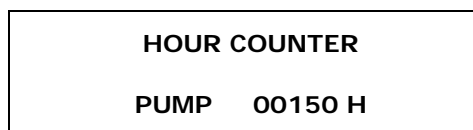


Fig. 5

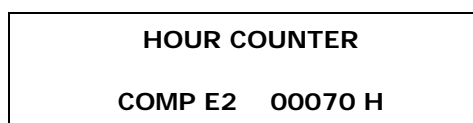


Fig. 6

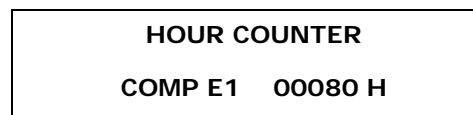


Fig. 7

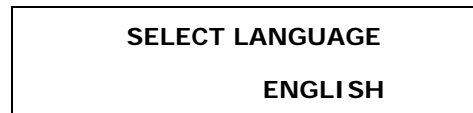
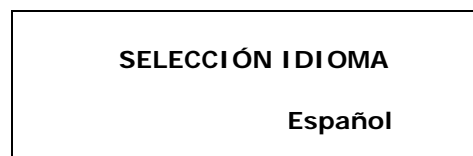
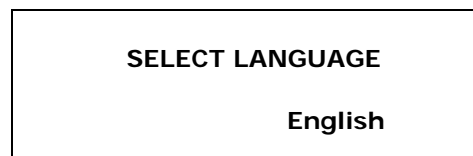


Fig. 8

### Language change

This option allows changing the screen language. There are 4 possibilities: Spanish, English, French and German. By means of the ENTER key, languages will change. To fix one, press the UP/DOWN keys.



## 7.6. ALARMS

### 7.6.1. VACUUM SYSTEM ALARM

If, when the vacuum pump is running, the vacuum level falls below 10 mbar in less than 4 minutes, the pump is stopped and the following message appears:

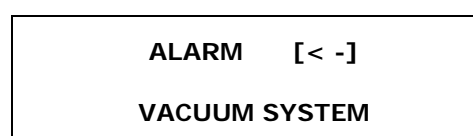


Fig. 9

Check all seals. Verify that valves and drain are closed.



Once the leak has been found the message will disappear by pressing the ENTER key. The vacuum pump will run again.

If the alarm does not disappear, contact the Technical Service Dept.

#### **7.6.2. VACUUM PROBE ALARM**

If the vacuum reading falls below  $1 \times 10^{-3}$  mbar, this shows that the vacuum gauge is broken or disconnected. The following message will appear:

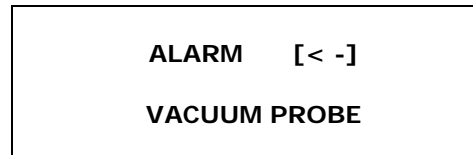


Fig. 10

Review the probe and replace it in case is broken. Once it is verified, connect again by pressing the ENTER key.

#### **7.6.3. TEMPERATURE PROBE ALARM**

If the temperature probe is broken or disconnected, the following message will appear:

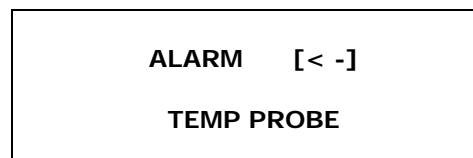
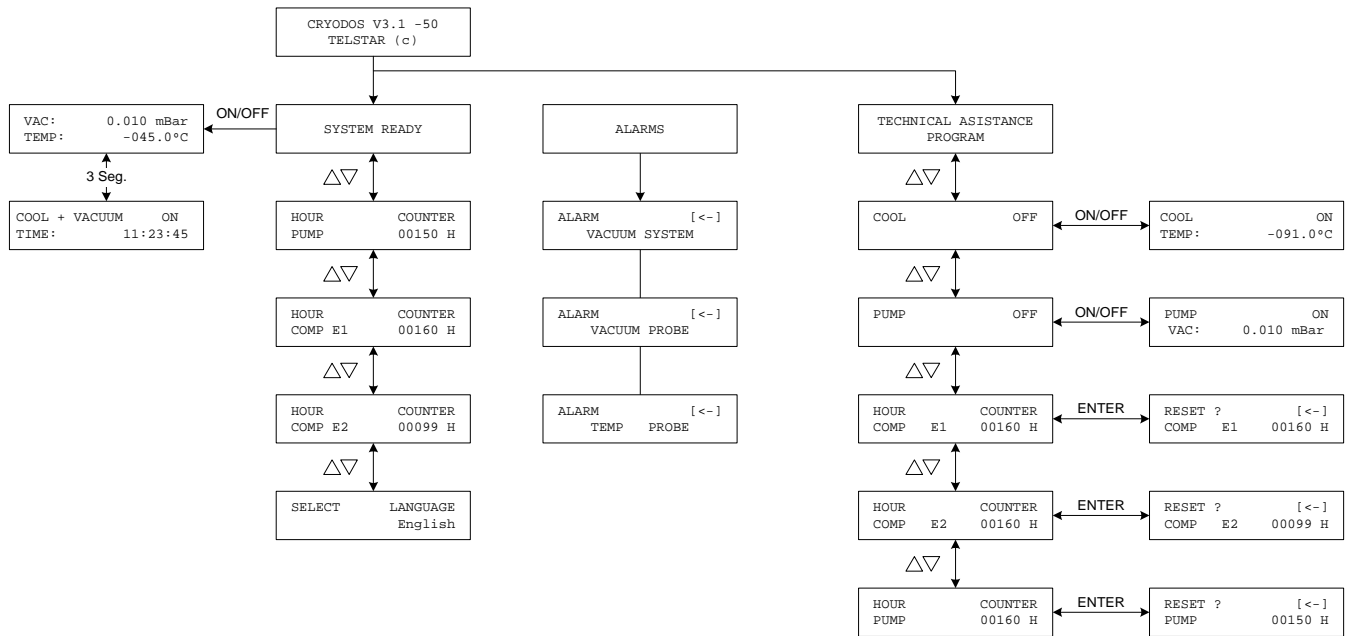


Fig. 11

Review the probe and replace it in case is broken. Once it is verified, connect again by pressing the ENTER key.

## 8. ATTACHED

### 8.1. WORKING PRINCIPLE



CRYODOS V3.1

## **8.2. GUARANTEE CERTIFICATE**

TELSTAR INDUSTRIAL, S.L. guarantees the replacement FREE of CHARGE of all the pieces suffering breakdown in the period of ONE YEAR, starting in the date of the expedition of the equipment.

The replacement of the pieces in guarantee can not in any case extend or modify the date of it.

TELSTAR INDUSTRIAL, S.L. will send in carriage forward and FREE of CHARGE the pieces to be replaced, against the reception of the defective pieces. The necessary work to be done for its substitution will be paid by the customer.

If the substitution of the pieces would take place in TELSTAR INDUSTRIAL, S.L. facilities, the client will pay the transport of the unit.

This equipment has undergone satisfactorily all the tests and revisions. Therefore, any damage caused by transport or wrong installation is excluded of this guarantee.

This guarantee does not include any of the fungible materials comprised in the equipment, such as filters, fluids, filaments, etc.

The inappropriate use of this equipment provokes the guarantee to be void. This involves the exclusion of any liability in concept of indemnity for eventual direct or indirect damages of any nature, either material or personal.

Likewise, this guarantee will be void if pieces other than the ones supplied by TELSTAR INDUSTRIAL, S. L. are used for the repairing of the equipment.

**NOTE. This guarantee is valid for all the equipment unless otherwise stated. In order to obtain any information about this guarantee, please contact with our Quality Assurance Department.**

### 8.3. CE CERTIFICATE



#### DECLARACIÓN DE CONFORMIDAD

#### DECLARATION OF CONFORMITY

Este certificado se emite para el aparato eléctrico/ *This certificate is issued for the electrical apparatus:*

#### LIOFILIZADORES DE LABORATORIO E INVESTIGACIÓN

#### LABORATORY AND RESEARCH FREEZE DRYERS

MODELOS/ *MODELS*: CRYODOS-50, CRYODOS-80

Fabricada y sometida a Certificación por **TELSTAR INDUSTRIAL, S. L.**/ *Manufactured and submitted for Certification by TELSTAR INDUSTRIAL, S. L.*

**91/368/EEC** y **93/68/EEC** Directivas Seguridad en Máquinas/ *Machinery Safety Directives*.  
Normativa respecto a la cual se declara conformidad/ *Standard(s) to which conformity is declared:*

**EN 292-1, EN 292-2, EN 294, EN 418, EN 349**

**73/23/EEC** Directiva Seguridad Eléctrica/ *Electrical Safety Directive*.

Normativa respecto a la cual se declara conformidad/ *Standard(s) to which conformity is declared:*

**EN 61010-1** (Requisitos de seguridad de equipos eléctricos de medida, control y uso en laboratorio/ *Safety requirements for electrical equipment for measurement, control and laboratory use*).

Directiva **89/336/EEC** Compatibilidad Electromagnética/ *Electromagnetic Compatibility Directive*.

Normativa respecto a la cual se declara conformidad/ *Standard(s) to which conformity is declared:*

**EN 50082-1, EN 55022, EN 61000-3-2**

El año de marcaje CE fue en 1997/ *The CE marking was affixed in 1997.*

Por la presente, **TELSTAR INDUSTRIAL** declara que el equipo arriba especificado cumple con las Directivas y Normativas arriba mencionadas, siempre y cuando la instalación sea acorde a las especificaciones del fabricante.

**TELSTAR INDUSTRIAL** hereby declares that the equipment specified above conforms to the above Directives and Standards, when installed in accordance with the manufacturers specifications.



**Eva Perelló**  
**Quality Assurance**  
Terrassa July 2005