

User's Manual

FreeZone[®] Triad[™] Freeze Dry System

Models

7400030

7400040

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*Protecting your
laboratory environment*

LABCONCO[®]

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Please read the User's Manual before operating the equipment.

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Warranty

Labconco provides a warranty on all parts and factory workmanship. The warranty includes areas of defective material and workmanship, provided such defect results from normal and proper use of the equipment.

The warranty for all Labconco products will expire one year from date of installation or two years from date of shipment from Labconco, whichever is sooner, except the following;

- Purifier® Delta® Series Biological Safety Cabinets and PuriCare® Lab Animal Research Stations carry a three-year warranty from date of installation or four years from date of shipment from Labconco, whichever is sooner.
- SteamScrubber® & FlaskScrubber® Glassware Washers carry a two-year warranty from date of installation or three years from date of shipment from Labconco, whichever is sooner.
- Blood Drawing Chairs carry a ten year warranty.
- Carts carry a lifetime warranty.
- Glassware is not warranted from breakage when dropped or mishandled.

This limited warranty covers parts and labor, but not transportation and insurance charges. In the event of a warranty claim, contact Labconco Corporation or the dealer who sold you the product. If the cause is determined to be a manufacturing fault, the dealer or Labconco Corporation will repair or replace all defective parts to restore the unit to operation. Under no circumstances shall Labconco Corporation be liable for indirect, consequential, or special damages of any kind. This statement may be altered by a specific published amendment. No individual has authorization to alter the provisions of this warranty policy or its amendments. Lamps and filters are not covered by this warranty. Damage due to corrosion or accidental breakage is not covered.

Returned or Damaged Goods

Do not return goods without the prior authorization from Labconco. Unauthorized returns will not be accepted. If your cabinet was damaged in transit, you must file a claim directly with the freight carrier. Labconco Corporation and its dealers are not responsible for shipping damages.

The United States Interstate Commerce Commission rules require that claims be filed with the delivery carrier within fifteen (15) days of delivery.

Limitation of Liability

The disposal and/or emission of substances used in connection with this equipment may be governed by various federal, state, or local regulations. All users of this equipment are required to become familiar with any regulations that apply in the user's area concerning the dumping of waste materials in or upon water, land, or air and to comply with such regulations. Labconco Corporation is held harmless with respect to user's compliance with such regulations.

Contacting Labconco Corporation

If you have questions that are not addressed in this manual, or if you need technical assistance, contact Labconco's Customer Service Department or Labconco's Product Service Department at 1-800-821-5525 or 1-816-333-8811, between the hours of 7:00 a.m. and 6:00 p.m., Central Standard Time.

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Chapter 1:

Introduction

Congratulations on your purchase of a Labconco FreeZone® Triad™ Freeze Dry System, which is designed for laboratory lyophilization procedures. The refrigerant used in the refrigeration system is CFC-free so it will not endanger the environment. The unit is easy to install and maintain. Proper care and maintenance of this product will result in many years of dependable service.

Freeze Dry Process

Freeze drying is an important process in sample preparation and for the preservation and storage of biologicals, pharmaceuticals and foods. Of the various methods of dehydration, freeze drying (lyophilization) is especially suited for substances that are heat sensitive. Other than food processing (e.g., coffee, whole dinners), freeze drying has been extensively used in the development of pharmaceuticals (e.g., antibiotics) and preservation of biologicals (e.g., proteins, plasma, viruses and cell lines). The nondestructive nature of this process has been demonstrated by the retention of viability in freeze dried viruses and microorganisms.

Freeze drying is a process whereby water or other solvent is removed from frozen material by converting the frozen water directly into vapor without the intermediate formation of liquid water. The basis for this sublimation process involves the absorption of heat by the frozen sample in order to vaporize the ice; the use of a vacuum pump to enhance the removal of water vapor from the surface of the sample; the transfer of water vapor to a collector; and the removal of heat by the collector in order to condense the water vapor. In essence, the freeze dry process is a balance between the heat absorbed by the sample to vaporize the ice and the heat removed from the collector to convert the water vapor into ice.

Freeze Dry Rates

The efficiency of the freeze drying process is dependent upon the surface area and the thickness of the sample, the collector temperature and vacuum obtained, the eutectic point and solute concentration of the sample. It is important to remember these factors when trying to obtain efficient utilization of your freeze dry system. A listing of selected materials and their approximate drying times are shown in Table 1 for your reference.

Table 1

Safe Temperature and Drying Times for Selected Materials			
Material 10mm Thick	Safe Temperature °C	Collector Temperature °C	Hours (Approx.)
Milk	-5	-40	10
Urea	-7	-40	10
Blood Plasma	-10 to -25	-40	16
Serum	-25	-40	18
Vaccinia	-30 to -40	-50	22
Influenza Vaccine	-30	-50	24
Human Tissue	-30 to -40	-50	48
Vegetable Tissue	-50	-80	60

*Total sample quantities are contingent on various Triad Freeze Dry System capacities.

Up to the point of overloading the system, the greater the surface area of the sample, and the faster the rate of freeze drying. By contrast, for a given surface area, the thicker the sample the slower the rate of freeze drying. This is based on the fact that the heat of sublimation is usually absorbed on one side of the frozen sample and must travel through the frozen layer to vaporize water at the other surface. In addition, as the sample is freeze dried, the water vapor must travel through the layer of dried material. The thicker the sample, the greater the chance that the dried layer may collapse which would cause an additional decrease in the rate of freeze drying.

The surface area and thickness of the sample can usually be ignored when each sample contains only a few milliliters. However, for larger volumes, the samples should be shell frozen to maximize the surface area and minimize the thickness of the sample. The volume of the freeze dry container should be two to three times the volume of the sample.

In order for lyophilization to occur, ice must be removed from the frozen sample via sublimation. The collector and the vacuum pump accomplish this. The collector, which should be at least 15 to 20°C colder than the eutectic temperature (melting temperature) of the sample, traps vapor as ice. Since the vapor pressure at the collector is lower than that of the sample, the flow of water vapor is from the sample to the collector.

Since this vapor diffusion process occurs very slowly under normal atmospheric conditions, a good vacuum is essential to maintain an efficient rate. In many applications, the maintenance of a vacuum of 0.133 mBar or less is recommended.

The rate of freeze drying is directly proportional to the vapor pressure and the vapor pressure is dependent upon both eutectic temperature and solute concentration of the sample. For example, a solution of sodium chloride and water would freeze dry at a slower rate than pure water. The eutectic temperature of a sodium chloride solution is about -21°C and at this temperature the vapor pressure is about 1/16 that of water at 0°C . Although the eutectic temperature is not dependent upon the concentration of sodium chloride, the vapor pressure of the water would decrease as the concentration of sodium chloride increased. This is due to the fact that as the solute concentration increases, less of the surface area of the frozen sample is occupied by water. In general, most solutions or biological samples will have a eutectic temperature of -10° to -25°C . However, if the sample contains a simple sugar such as glucose or if the sample is animal or plant tissue, the eutectic temperature may be as low as -30° to -50°C .

Freeze Dry Capacity

The volume of a sample that can be freeze dried at one time is related to factors discussed previously and the size and design of the freeze dry system. With any given instrument, the capacity is based on the surface area of the sample, the eutectic temperature and concentration of the sample and the rate and amount of heat transferred to the frozen sample. Of these factors, the eutectic temperature is the most important factor in determining the amount of sample that can be freeze dried at one time, particularly when flasks are used. This is because as the eutectic temperature decreases, the vapor pressure decreases but the rate of heat absorption by the sample does not change. This tends to promote melting of the sample, which leads to a marked increase in vapor pressure and ultimately overloads the collector and vacuum pump. Samples that have eutectic temperatures of -20°C or lower should be placed on the freeze dry system one flask at a time so that the vacuum in the system may recover before adding another sample to the system. If the vacuum does not recover, the capacity of the freeze dry system has been exceeded and the sample should be removed.

If there is a problem with a particular type of sample melting when placed on the freeze dry system, dilution of the sample with more water or providing some insulation around the flask to decrease the rate of heat absorption by the sample may help.

Samples Containing Volatile Substances

In certain cases the solvent in a sample to be freeze dried may contain volatile components such as acetonitrile, methanol, acetic acid, formic acid or pyridine.

In addition to these substances having an effect on the eutectic temperature, they may increase the vapor pressure at the surface of the sample. Also, compared to water, they will require the absorption of less heat for sublimation to occur. Hence, samples that contain volatile substances will have a greater tendency to melt, particularly when placed in flasks or exposed to room temperature. If a sample containing a volatile substance tends to melt when placed on a freeze dry system, dilution of the sample with more water will help keep the sample frozen. For example, a 0.2M solution of acetic acid is much easier to freeze dry than a 0.5M solution.

Chapter 2: Prerequisites

Before you install your Triad Freeze Dry System, you must be certain that the area is level and of solid construction. An electrical source must be located near the installation site.

Carefully read this chapter to learn:

- Electrical supply requirements.
- Location requirements.
- Vacuum pump requirements.

Refer to Appendix C: Freeze Dry System Specifications for complete electrical and environmental conditions, specifications and requirements.

Electrical Requirements

The Triad Freeze Dry System requires a dedicated electrical outlet. This outlet requires a 20 Amp circuit breaker or fuse for all models. The power cord on 230V models is equipped with a NEMA 6-20P plug. If this does not match with the available receptacle, remove this plug and replace it with an approved plug of the suitable style.

Location Requirements

The Triad Freeze Dry System should be located in an area that provides an unobstructed flow of air around the cabinet. This air cools the refrigeration system. The refrigeration system draws air in through the left side and exhausts it through the right side. A minimum of 3" must be allowed between the rear and both sides of the Triad Freeze Dry System and adjacent wall surfaces. Restriction of airflow during operation could adversely affect performance. The Triad Freeze Dry System must be placed on a work surface or table that will support over 400 lbs.

Refer to Appendix B: Freeze Dry System Dimensions for drawings.

Vacuum Pump Requirements

The user must provide a vacuum pump. A vacuum pump with a displacement of 144 liters per minute and 0.0002 mBar ultimate pressure is adequate for most samples. The inlet fitting on the vacuum pump must be suitable for 3/4" ID vacuum hose, which is provided with the FreeZone[®] Triad Freeze Dry System. It is recommended that the vacuum pump is equipped with an exhaust filter to minimize oil mist exhausting from the vacuum pump.

The operating vacuum level may be set on the freeze dry system. The higher the pressure is set, the more likely it is that oil mist will be exhausted.

Vacuum pumps should be equipped with a reverse IEC plug. This will allow the vacuum pump to be plugged into the receptacle on the back panel of the Freeze Dry System. Refer to Appendix D: Triad Freeze Dry System Accessories for vacuum pumps available from Labconco.

Chapter 3: Getting Started

Now that the site for your Triad Freeze Dry System is properly prepared, you are ready to unpack, inspect, install and test your freeze dry system.

Read this chapter to learn how to:

- Unpack and move your freeze dry system.
- Set up your freeze dry system.
- Safely use solvents with your freeze dry system.

Unpacking Your Triad Freeze Dry System

Carefully unpack your freeze dry system and inspect it for damage that may have occurred in transit. If your freeze dry system is damaged, notify the delivery carrier immediately and retain the entire shipment intact for inspection by the carrier.

The Triad Freeze Dry System weighs over 400 lbs. (181 Kg). The carton allows for lifting with a mechanical lift truck or hand truck. If you must lift the freeze dry system manually, use at least four (4) persons and follow safe lifting guidelines.

If your freeze dry system was damaged in transit, you must file a claim directly with the freight carrier. The United States Interstate Commerce Commission rules require that claims be filed with the delivery carrier within fifteen (15) days of delivery. Labconco Corporation and its dealers are not responsible for shipping damage.

Do not return goods without the prior authorization of Labconco, unauthorized returns will not be accepted.

Do not discard the carton or packing material for your freeze dry system until you have checked all of the components, installed and tested the system.

Triad Freeze Dry System Components

Locate the model of freeze dry system you received in the following table. Verify that the components listed are present and undamaged.

If you did not receive one or more of the components listed, contact Labconco Corporation immediately for further instructions.

Catalog #	Product Description
7400030	Triad Freeze Dry System – 230V, 50 Hz
7400040	Triad Freeze Dry System – 230V, 60 Hz

Plus the following:

Part #	Qty.	Component Description
7373300	1	User's Manual
1342100	1	Power Cord – 230V
7373436	1	Tubing
1488800	2	Clamp
1291000	1	Female IEC Plug

Setting Up Your Triad Freeze Dry System

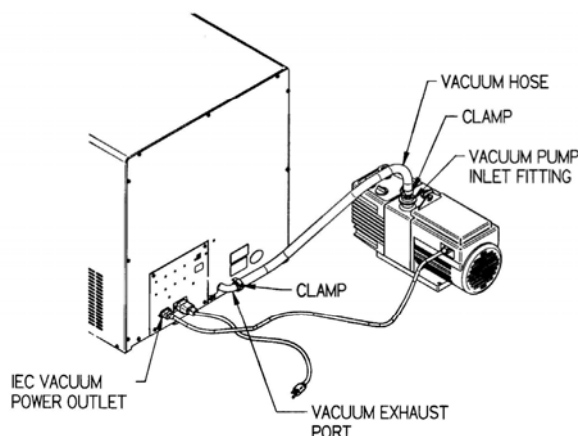
Component Orientation and Hose Connections

A minimum of 3" should be allowed between the back of the freeze dry system and the adjacent wall surface and between the sides of the Freeze dryer and the adjacent wall surfaces. Restriction of the airflow through the cabinet during operation could adversely affect performance.

Position the vacuum pump near the vacuum exhaust port on the rear of the cabinet. Attach the supplied hose to the exhaust port using the clamp provided. Attach the other end of the hose to the inlet fitting on the vacuum pump. Secure with the clamp provided. The hose may be cut to proper length to allow for gentle bends without kinks.

Connect the power cord from the vacuum pump into the outlet on the back of the unit. If your vacuum pump power cord does not have a female IEC plug, replace the cord end with the female IEC plug provided with the unit (Part number 1291000).

Figure 1



Venting the Vacuum Pump

If any materials will be placed in the freeze dry system that can liberate hazardous gases when heated, the vacuum pump exhaust must be vented to a fume hood or other ventilation device.

Electrical Connection

Connect the power cord into the receptacle on the back of the freeze dry system and connect the other end into a suitable power receptacle.

The freeze dry system is now installed and must be tested to make certain the system is free of leaks. Close the door, turn on the freeze dry system refrigeration and allow the collector temperature to reach -40° or lower, this could take 20 to 30 minutes. Make sure the Stoppering Control is in the “RAISED” position and the Vacuum Release control is in the “CLOSED” position. Start the vacuum pump and monitor the vacuum gauge. The vacuum on the freeze dry system should reach 0.133 mBar within 30 minutes and should achieve an ultimate vacuum of 0.040 mBar or lower within 18 hours. If 0.040 mBar cannot be achieved, consult the troubleshooting section of this manual.

Chemical Resistance of Freeze Dry System Components

The FreeZone[®] Triad Freeze Dry System is designed to be chemically resistant to most compounds that are commonly used in Freeze drying processes. However, by necessity, the freeze dry system is comprised of a number of different materials, some of which may be attacked and degraded by certain chemicals. The degree of degradation is dependent on the concentration and exposure duration.

Some of the major components of the freeze dry system that are susceptible to degradation are as follows:

Component	Material	Acids			Buffers			Solvents					
		Acetic Acid 20%	Formic Acid	Trifluoroacetic Acid (TFA)	Calcium Chloride	Sodium Phosphate	Acetone	Acetonitrile	Carbon	Cyclohexane	Dioxane	Methyl t-Butyl Ether (BTBE)	Pyridine
Valve Stem	Acetal (Delrin)	C	D	D	D		D						
Door	Acrylic			D			D	D	D				
Hoses, Gaskets & Valve Bodies	Neoprene	C	D	D			C	C	D	D	D	C	D
Flask Top, Drain Hose	Silicon Rubber		C	D		D			D	D	D	C	D
Chamber & Fittings	Stainless Steel				C								

C = Moderate degradation; limited use

D = Severe degradation; infrequent use recommended; immediate thorough cleaning required.

- Most common compounds used in freeze drying processes, if allowed to enter the vacuum pump, will degrade the oil and cause damage to the vacuum pump.
- Sugars and proteins typically will have minimal negative effect on any of the materials of construction.

When using compounds in the freeze dry system that are hostile to the materials of construction, it is imperative the equipment is thoroughly cleaned after use.

- Rubber and plastic components that have been exposed to damaging compounds should be removed and flushed with water.
- The oil in the vacuum pump should be checked often. It must be changed if it is cloudy, shows particles or is discolored. The useful life of vacuum pump oil can be extended if the vacuum pump is operated for an extended period of time after a freeze dry run. This allows contaminants to be purged from the hot oil. This must be done with the inlet to the pump blocked off to prevent air from free flowing through the pump. This is accomplished by closing all sample valves on a clean, dry, freeze dry system and turning on the vacuum pump. If the pump is operated at an elevated vacuum level ($> 10\text{mBar}$), oil may be expelled from the pump and damage could occur.

Optional secondary traps are available to help extend the life of the vacuum pump. These traps are installed between the freeze dry system and the vacuum pump. Contact Labconco for assistance in determining which trap is right for your application.

With prudent maintenance the freeze dry system will provide years of service. Warranty on the affected parts will be voided if maintenance has been obviously neglected. If you have questions about using specific compounds in the freeze dry system, contact Labconco Technical Service at 1-800-821-5525 or 816-333-8811 or e-mail: labconco@labconco.com.

Solvent Safety Precautions



Solvents used in the freeze dry system may be flammable or hazardous to your health. Use extreme caution and keep sources of ignition away from the solvents. When using flammable or hazardous solvents, the vacuum pump must be vented to a fume hood.

Hazardous materials such as strong acids or bases, radioactive substances and volatile organics must be handled carefully and promptly cleaned up if spilled. If a sample is spilled in the collector chamber it must immediately be cleaned up per MSDS procedures or damage to the equipment or injury may occur.

NOTE: Various Federal, State or local regulations may govern the disposal of substances used in connection with this equipment. All users of this equipment are urged to become familiar with any regulations that apply in the user's area concerning the dumping of waste materials in or upon water, land or air and to comply with such regulations.

Chapter 4: Operating Instructions

Read this chapter to learn how to:

- Understand the display.
- Operate the controls.
- Connect samples.

Do not use the freeze dry system in a manner not specified by the manufacturer (refer to Appendix C: Freeze Dry System Specifications). The electrical protection may be impaired if used inappropriately.

Triad Freeze Dry System Controls

The control panel for the Triad Freeze Dry System is shown with a description about its function.

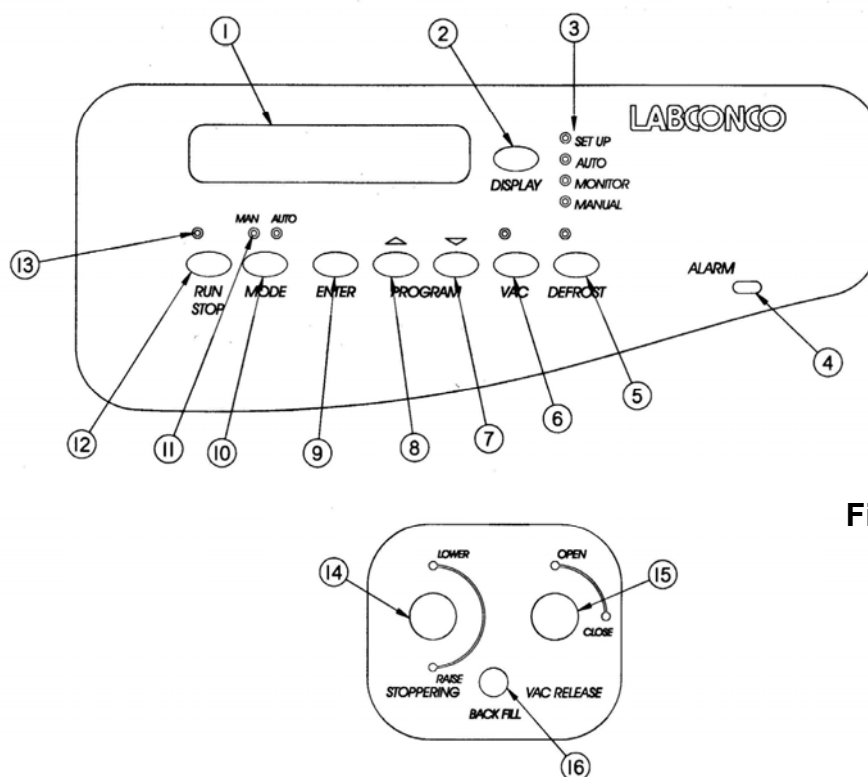


Figure 2

1. **Display:** Displays all necessary programming and operational data. Provides prompts to aid in programming.
2. **Display Button:** Changes format of display from “monitor” to “auto” to “manual” to “set up” screen.
3. **Display Indicators:** Green INDICATOR shows which display “screen” is shown.
4. **Alarm Indicator:** Red INDICATOR shows that a system alarm has occurred.
5. **Defrost:** Used to manually start or stop the defrost operation.
6. **Vacuum:** Used to manually start or stop the vacuum pump.
7. **Decrease Button:** Used in programming to decrease a parameter set point.
8. **Increase Button:** Used in programming to increase a parameter set point.
9. **Enter Button:** Used in programming to enter a selected set point into memory.
10. **Mode Selector Button:** Selects the mode of operation – either manual or automatic.
11. **Mode Indicator:** Green INDICATOR shows whether the control is set to operate either in the automatic or manual mode.

12. **Run/Stop Button:** Initiates the start or stop of the lyophilization process.
13. **Run/Stop Indicator:** Green INDICATOR burns steadily while freeze drying is in progress and turns off when the Run/Stop button terminates a run. The green INDICATOR flashes if freeze drying is in progress and a power failure occurs.
14. **Stoppering:** Controls the stoppering platen up and down movement. The mechanism will function only while the chamber is under vacuum. The stoppering action should be closely monitored when operating this control.
15. **Vacuum Release:** Vents the chamber so the chamber door can be opened. This control can also vent gas into the chamber when the gas supply is properly connected to the backfill port.
16. **Back Fill:** A regulated tank of gas may be connected to the port to allow the introduction of gas into the chamber after freeze drying. The port accepts 1/8" tubing.



CAUTION

Ethylene Oxide is not recommended for use in this freeze dry system for decontamination because of its hazardous and corrosive properties.

17. **Power Switch:** Turns all power to Triad Freeze Dry System ON or OFF. (Not shown). Located on left side of cabinet.

Operation Checklist

The following checklist should be followed prior to each use of your freeze dry system:

1. Wipe out the interior of the chamber with a soft cloth or paper towel to remove any moisture or debris.
2. Check the collector tray drain hose to ensure that the hose is free of moisture and that the drain plug is securely installed.
3. Using a soft, lint-free cloth or paper towel, wipe the door gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the door gasket.
4. Check that each sample valve is closed or in the “vent” position. See Figure 4.

Vacuum Pump Ballast Setting

Most vacuum pumps are equipped with gas ballast mechanism. The freeze dry process requires high vacuum. Therefore, it is recommended that the gas ballast be closed during the operation of the Triad Freeze Dry System.

Note: If the gas ballast is left open for extended periods of operation, the oil can be pumped out the exhaust, causing the pump to fail.

Operating the Triad Freeze Dry System

Set-Up

Vacuum may be displayed in mBar, Pascal (Pa) or Torr and the temperature may be displayed as either °F or °C. The refrigeration system and vacuum pump operating time may be monitored and the operating vacuum level may be set. To configure your Triad Freeze Dry System, turn the main Power Switch ON. Press the DISPLAY button until the SET UP indicator is lit.

The display will show:

VACUUM UNITS:		
MBar	Pa	Torr

- Press \uparrow or \downarrow until the desired units are flashing.
- Press ENTER.

The display will show:

VACUUM SET POINT:	
XXXX	YYY

Where YYY is the units selected above.

- Press \uparrow or \downarrow until the desired vacuum operating level is displayed. See “Setting the Operating Vacuum Level” for guidelines.
- Press ENTER.

The display will show:

TEMPERATURE UNITS:	
^C	^F

- Press \uparrow or \downarrow until the desired units are flashing.
- Press ENTER.

The display will show:

REFRIG TOTAL HOUR: XXXX
SERVICE HOUR: XXXX

- Press ENTER if you do not want to reset SERVICE HOURS.
- Press and hold ↵ for 5 seconds to reset the Service Hours to 0. This allows you to keep track of the time the refrigeration system operated since it was serviced.

The display will show:

REFRIGERATION TOTAL HOURS: XXXX:	
RESET SERVICE HOUR: ?	X

- Press ENTER.

The display will show:

VACUUM TOTAL HOURS: XXXX:	
SERVICE HOUR: XXXX	

- Press ENTER if you do not want to reset SERVICE HOURS.
- Press and hold ↵ for 5 seconds reset the Service Hours to 0. This allows you to keep track of the time the vacuum pump operated since it was serviced.

The display will show:

VACUUM TOTAL HOURS: XXXX:	
RESET SERVICE HOUR: ?	X

- Press ENTER.

The display will show:

RS-232 TRANSMISSION RATE:	
10	SECONDS

- Press ↑ or ↓ until the desired time interval is shown. The time between data transmission may be set to occur at 10, 30, 60, 300 or 600 seconds intervals.
- Press ENTER.

The display will show:

VACUUM UNITS:		
MBar	Pa	Torr

- At any time the DISPLAY button can be pressed to select a different function.

Setting the Operating Vacuum Level

The vacuum level may be manually set by the user to optimize the freeze dry process. Normally, the sublimation rate will increase if there is less vacuum (a higher pressure) in the Triad Freeze Dry System. A good starting place is to set the vacuum to a level that will hold the sample temperature approximately 10°C cooler than the eutectic temperature. Adjustments to the vacuum level must be made for various freeze drying conditions. Factors that must be considered are whether the sample is freeze dried on heated shelves or in glassware attached to manifold valves, the volatility of the sample itself, the size of the sample and the heat energy supplied to the sample. When the vacuum control is set to operate at less vacuum, the ice holding capacity of the collector may be decreased.

Some guidelines for setting the Triad Freeze Dry System are shown below. These show pre-freezing temperature and vacuum levels. The user of the specific samples that are being freeze dried must determine exact protocols.

Material	Solidification/Eutectic Temperature	Pre-Freeze Temperatures	Vacuum Set Point
Bacteria, Virus	-40°C and Colder	-50°C and Colder	0.404 mBar and Lower
Milk	-5 to -13	-15 to -23	1.65 to 0.77
Fungi	-40 and Colder	-50 and Colder	0.04 and Lower
Vegetable Tissue	-25 to -50	-35 to -60	0.22 to 0.01
Human Tissue	-30 to -40	-40 to -50	0.12 to 1.04
Blood Plasma	-10 to -25	-20 to -35	1.03 to 0.22
Vaccine	-30 to -40	-40 to -50	0.12 to 0.04

Manual Control Operation

The manual operation has three selectable modes: Pre-Freeze, In-Flask and controlled shelf temperature. The Pre-Freeze setting allows the shelf temperature to freeze samples below -55°C obtaining “Max Cold” temperature of approximately -75°C . The In-Flask setting is used to freeze dry pre-frozen samples when using only the sample valves on the left side of the unit. There is no shelf temperature control in this mode. When using the controlled shelf set point temperature setting, the shelf temperature will maintain the set point selected between -55°C to 50°C . This mode should be used when freeze drying samples on the shelf. A flask can also be added to the sample valves in this mode.

- (1) Turn the Power Switch ON. The display will become active.
- (2) Push the DISPLAY button until the MANUAL indicator lights. This will cause the manual display screen to be shown with the previously entered set point temperature or operational mode.

SET POINT TEMP -30°C

The temperature units are displayed in the units ($^{\circ}\text{F}$ or $^{\circ}\text{C}$) selected in the Set Up.

The temperature may be set to control the shelf anywhere from $+50^{\circ}\text{C}$ to -55°C in 1°C increments. To pre-freeze samples below -55°C set temperature to “Pre-Freeze.” To freeze dry samples using the manifold sample valves change set point temperature to: “In-flask.”

- (3) If the set point or operating mode needs to be changed, press the INCREASE or DECREASE button until the desired set point temperature or mode is displayed.
- (4) Press ENTER. If ENTER is not pressed within 10 seconds, the set point will revert to the previously entered parameter.
- (5) Press MODE button until the manual indicator lights.
- (6) Press RUN/STOP button. The RUN/STOP indicator will light and the refrigeration system and/or heater will operate.
- (7) If vacuum is required press VAC Button. Do not run vacuum with unfrozen samples in chamber.
- (8) To change the set point temperature in the middle of a run, repeat steps 2, 3 and 4. The system will adjust to the new temperature set point.
- (9) To monitor system operation, press DISPLAY until the Monitor indicator lights. The display will simultaneously show shelf (SHLF) temperature, collector (COL) temperature and the sample (SAMP) temperature. If the sample probe is not plugged into its jack, the SAMP temperature display will show “- -”. If in the Pre-freeze mode the (COL) display will show “- -”. If in the In-flask mode the (SHLF) the display will show “- -”.

The segment numbers will always show “M” while operating in the manual mode. Segment numbers are used only in the automatic mode to identify portions of the programmed cycle. Temperatures and vacuum are displayed in the units selected during the set up procedure. Above 5.0 mBar the display will show “HIGH” vacuum.

SHLF=XXX	SAMP=XXX °C
COL= XXX	SEG =M VAC = HIGH m

SHLF	Shelf temperature
SAMP	Sample probe (if probe is not installed, the display shows “ -”)
C	°C
F	°F
COL	Collector Temperature
SEG	Shows what segment is active and ramping or holding.
M	Manual mode
VAC	System vacuum level
m	mBar
P	Pascal
T	Torr

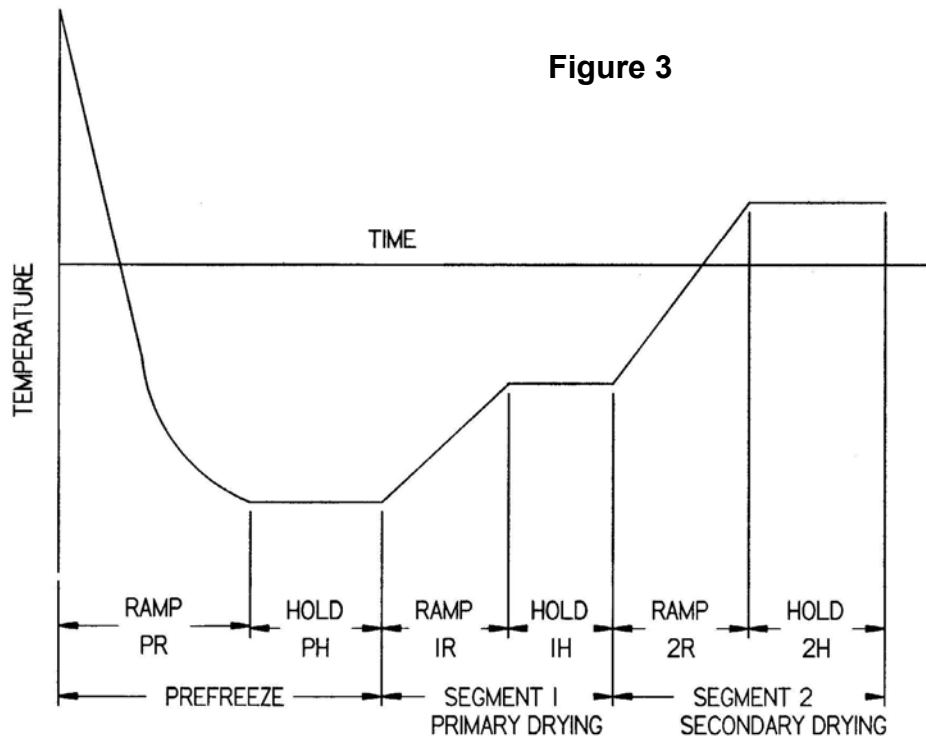
- (10) To stop, push RUN/STOP button, which will turn off the refrigeration system and the heater. To turn off the entire system, turn the Power Switch OFF.

Automatic Control Operation

The Triad Freeze Dry System is equipped with a microprocessor-based controller that permits temperature to be programmed using as many as six different segments. The first segment is Pre-freeze with automatic “Max Cold” setting and an adjustable time setting of 0, 3 hours to 99.9 hours or indefinite. The additional five segments consist of a temperature ramp function and a temperature hold function. There are five programs of six segments each that can store settings. One of the five may be selected by pressing the DISPLAY button until the AUTO indicator lights. Press the UP/DOWN buttons to select the desired program, and then press ENTER. The display will change to show the parameters for this program. The ramp allows the temperature of the sample to be increased or decreased at any desired rate within the capacity of the heating and cooling systems of the Triad Freeze Dry System.

The Triad Freeze Dry System without samples on shelves is capable of cooling at a rate of approximately .5°C/minute from 50°C to 0°C, .25°C/minute from 0°C to –55°C. and can heat at approximately 3°C/minute. Hold temperatures may be set anywhere from +50°C to –55°C in 1°C increments. Hold times are set in hours.

When the desired temperature is achieved, the hold function will maintain that temperature for the programmed length of time. The microprocessor control has a built in memory of the last entered program to allow the identical protocol to be repeated by simply pushing the RUN button. A typical 3-segment program is shown in Figure 3.



Programming

The Triad Freeze Dry System will store up to five programs numbered 1 through 5. The five programs, once programmed, will be retained in memory. To program a run, select the program number that you want to use and enter the parameters as follows.

Turn the Power Switch to the ON position. Press the DISPLAY button until the AUTO Indicator lights.

NOTE: Values shown in this example are for reference only.

The “P” in the display will flash and the program number that will be run is shown. To select different programs, press the UP or DOWN button to change program number (Programs 1 through 5). When the desired program number is displayed, press ENTER.

The display will show the parameters that were last entered for this program.

P1	SEG 1	RAMP	00.0°C/MN
HOLD	-34°C	TIME	00.0

If any of the parameters are changed, the ENTER button must be pressed for the new value to be stored. If ENTER is not pressed within 10 seconds after the last parameter adjustment, the value will return to the previously stored value. The flashing parameter name shows the parameter that will be changed. Pressing ENTER only will advance to the next parameter.

After the AUTO RUN cycle is started, segments with a value greater than the segment running may have their parameters changed. Only the selected program may be modified. To modify a different program, the system must be stopped and the desired program selected.

Starting the Freeze Dry Cycle

After the desired cycles have been programmed, push the MODE button to select AUTO. Push the RUN/STOP button to start the program running. The RUN/STOP indicator will light. The system display will change and the display will change to MONITOR mode.

During the pre-freeze segment the status display will be as follows:

P1 SHLF = -70	SAMP= -70°C
COL = ---	SEG = PR VAC = hi

P1	Selected Program
SHLF	Shelf Temperature
SAMP	Sample Temperature (if probe is not installed, the display shows ---)
°F or °C	Temperature Units
COL	No temperature displayed
SEG	Pre-Freeze Mode R(Ramp) H(Hold)
VAC	System Vacuum Level is above 5 mBar

During the ramp cycle of the active segment, the status display will be as follows:

P1 SHLF = -55	SAMP= -55°C
COL = -85	SEG = 1R VAC = .03M

P1	Selected Program
SHLF	Shelf Temperature
SAMP	Sample Temperature (if probe is not installed, the display shows ---)
°F or °C	Temperature Units
COL	Collector Temperature
SEG	Shows what segment is active and Ramping (R)
VAC	System vacuum level
M	MBar
P	Pascal
T	Torr

During the hold cycle of the active segment the display will alternate as follows:

P1 SHLF = -55	SAMP= -55°C
COL = -85	SEG = 1H VAC = .030M

P1 SHLF = -55	SAMP= -55°C
COL = -85	TR = 4.6 VAC = .030M

P1	Selected Program
SHLF	Shelf Temperature
SAMP	Sample Temperature (if probe is not installed, the display shows ---)
COL	Collector Temperature
SEG	Shows what segment is active and holding (H)
TR	Shows time remaining segment. If time is 10.0 hours or more the time is displayed in 1/10 hours. If time is less than 10.0 hours, the time is displayed in 1/100 hours. If the hold time was programmed to be indefinite, the display will show INDF.
VAC	System vacuum level
M	MBar
P	Pascal
T	Torr

Changing the Program Parameters in the Middle of a Run

- (1) Press the DISPLAY button to obtain Monitor screen. Note the current operating segment.
- (2) Press the DISPLAY button to obtain Auto screen.
- (3) By pressing the up button enter a segment number that follows the current segment. The current segment cannot be modified while the system is running. Press ENTER
- (4) Modify as required.
- (5) Press ENTER.

Stopping the Freeze Dry Cycle

1. At the end of the last programmed segment, the freeze dry system will automatically stop. The refrigeration system, the heater and the vacuum pump will remain on. The display will show Segment "E" (End). To shut off, press RUN/STOP button.
2. To stop before the completion of the last programmed segment, press RUN/STOP. The RUN/STOP indicator will turn off, as will the refrigeration system, heater and vacuum pump. The display will remain active but time functions will stop operating.
3. Pressing the Power Switch will shut off the entire system.
4. If the power is shut off during the program, when the power is turned backed on the system will resume operation if the collector temperature has not risen above -30°C. The unit will then attempt to complete the programmed cycle from the point at which it was shut off. If the collector temperature is greater than -30°C the system will not resume operation. The RUN indicator and ALARM indicator will flash to indicate power loss during the programmed run.

Monitoring the System Operation

Press the DISPLAY button to obtain the Monitor Screen. The display will show the collector temperature, shelf temperature, system vacuum and sample temperatures. If the sample probe is not connected, the sample temperature display will show “- -”. The temperatures and vacuum are displayed in the units selected in Set Up. Above 5.0 mBar the display will show “hi” vacuum.

The ice collecting coil temperature and the vacuum level should be monitored. Higher than desired collector coil temperature or vacuum levels will inhibit or ruin the freeze dry process.

Sample Freezing

Before the freeze dry process can occur, the products to be dried must be in a frozen state. This can be accomplished in a freezer separate from the freeze dry system or on the shelf in the Triad Freeze Dry System. If the samples will be pre-frozen on the shelf inside the Triad, turn ON the Power Switch and set the controls to cool the shelf. This is done by setting the temperature to “pre-freeze” in the manual mode or the pre-freeze segment in the automatic mode can be programmed to cool the shelves and start the vacuum after the end of the pre-freeze segment. If desired, place the temperature probe in a sample vial. Monitor the shelf and sample temperatures by pressing the DISPLAY button until the monitor indicator lights.

Shelf Loading

The stoppering mechanism is capable of generating a very strong force, which can damage the shelf or mechanism. Therefore, it is important to distribute the serum bottles to be stoppered evenly across the entire surface of the shelf. When stoppering vials, always place a vial on each corner of the shelf.

Freeze Drying Inside the Chamber

The following procedure should be followed when freeze drying using the temperature controlled shelf inside the vacuum chamber.

Manual Mode with Pre-Frozen Samples

1. With the display set to manual, select the desired “set point temperature” between -55°C and 50°C .
2. Select MAN mode.
3. Start the refrigeration system by pressing “RUN/STOP.” Close the door; turn on the vacuum.
4. When the collector temperature reaches -80°C and the shelf temperature reaches the desired set point. Turn off the vacuum pump and release the vacuum. Place the frozen samples on the shelf. Close the door and turn the vacuum on.

Note: The system will go from room temperature start up to -55°C in under 7 hours.

5. Shelf temperatures and vacuum set points can be changed at anytime during the manual Freeze Dry process.

Manual Mode with Unfrozen Samples

1. With the display set in manual, select “Set Point Temperature” = Pre-Freeze.
2. Set mode to MAN.
3. Place unfrozen samples on the shelf and close the door. The sample probe can be placed in the sample.
4. Start the refrigeration system by pressing “RUN/STOP.”
5. Start the vacuum pump by pressing “VAC.” Let the pump run 10 seconds. If the pump is allowed to run too long, sample may be lost.
6. The shelf temperature will cool to approximately -75°C and freeze the sample.

Note: Starting the system at room temperature will take approximately 6 hours to freeze a 2 liter tray of water.

7. When the sample is frozen change the shelf “Set Point Temperature” to the desired temperature between -55°C and 50°C .
8. Turn on vacuum.
9. Shelf temperatures and vacuum set points can be changed at anytime during the manual freeze dry process.

Auto Mode with Pre-Frozen Samples

1. With the display set to Auto, program the desired ramp, temperature and hold times for each segment of the program. The “PREFREEZE” segment time should be set to 0. This allows the program to start at segment 1.
2. Set mode to AUTO.
3. Start the refrigeration system by pressing “RUN/STOP.” Start the vacuum pump.
4. When the collector temperature reaches -80°C and the shelf temperature reaches the desired set point of segment 1, turn off the vacuum pump and release the vacuum. Place the frozen samples on the shelf. Close the door and turn the vacuum on.

Auto Mode with Unfrozen Samples

1. With the display set to Auto, program the desired ramp, temperature and hold times for each segment of the program. The “PREFREEZE” segment time must be set between 3 hours and “INDEF.” This allows the shelf to cool and pre-freeze the sample. The samples must be completely frozen during “PRE FREEZE” or samples will be compromised and damage could occur to the vacuum pump when turned on in segment 1.

2. Set mode to AUTO.
3. Place the unfrozen samples on the shelf and close the door.
4. Start the refrigeration system by pressing "RUN/STOP."

Note: The vacuum pump will run 10 seconds and then turn off automatically.

Stoppering Vials

The stoppering operation (when desired) is performed after the freeze dry process is complete and before breaking vacuum. To seal vials, move the Stoppering control toward the "LOWER" position. This action allows the diaphragm above the stoppering platen to inflate causing the platen to lower. The platen will lower until it contacts the vials on the shelf. The rubber stoppers will be pressed into the vials. Monitor the stoppering process by looking through the chamber door.

When all of the vials appear to be stoppered, move the Stoppering Control to the "RAISE" position. This opens the diaphragm to the vacuum pump, which deflates the diaphragm. Opening the Vacuum Release Control also deflates the stoppering diaphragm. The Stoppering Control should be left in the "RAISE" position when not stoppering. The stoppering mechanism is equipped with anti-tilt switch which will stop the platen from lowering if it tilts more than 4°. If this happens return the stoppering control valve to "RAISE" position. This will deflate the diaphragm and allow the platen to return to the raised position. Determine the cause of the tilt condition before proceeding.

Vacuum Break/Backfilling

To open the chamber door, the vacuum must be released. To release the vacuum, move the Vacuum Release Control to the "OPEN" position and shut off the vacuum pump by pressing the VAC button. The vacuum can also be released more quickly by opening one of the sample valves.

Air enters the vacuum chamber through the Back Fill Port. When the sound of air through the Back Fill Port is no longer audible, the chamber door is ready to open.

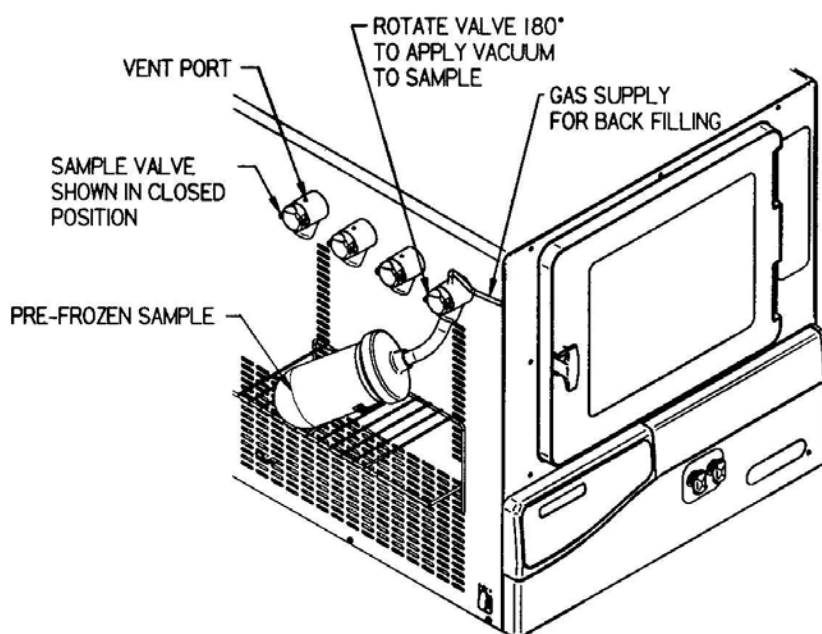
To backfill the chamber with a gas, connect a cylinder of the desired gas to the Back Fill Port. The port will accept 1/8" tubing. The bottle must be equipped with a regulator set to 15 psi maximum. The gas can be metered with the Vacuum Release Control and chamber pressure monitored with the freeze dry system's vacuum gauge. Allow the vacuum pump to run for a few minutes while the gas enters to permit the back fill gas to surround the freeze dried sample. When the vacuum pump is turned off, air will enter the system through the vacuum break valve located between the vacuum pump and the collector chamber.

Freeze Drying Using Manifold Valves

The following procedure should be followed when using the sample valves for the freeze dry process:

1. Turn on the Triad Freeze Dry System. Select manual mode, change the set point temp to “IN-FLASK” and turn on the vacuum.
2. Once the collector temperature is less than -80°C and vacuum is less than 0.133 mBar, connect a pre-frozen sample to a sample valve on the left side of the cabinet using an adapter. Turn the plastic valve knob to the “VACUUM” position to open the valve. The bevel on the knob should be positioned toward the sample port to apply vacuum to the sample. (see Figure 4).
3. Before adding another sample, allow system vacuum to return to the vacuum set point. Any combination of valves and sample sizes may be utilized at one time provided that the system vacuum and collector temperature remain sufficiently low to prevent melting of the frozen sample.
4. When all the frost has disappeared from the outer surface of the sample container and no cold spots can be detected by handling the container, primary drying is nearly complete. To be certain of low final moisture content, dry the sample for several hours past this point.

Figure 4



5. To remove a container after freeze drying is complete, turn the plastic knob on the valve to the “VENT” position, which closes the valve and vents the container. Should backfilling with an inert gas be required, connect the gas supply line to the vent port on the valve before turning the plastic knob on the valve to vent position. The sample container may now be removed. In the vent position the bevel on the valve knob should point away from the sample port.
6. Ampules may be flame sealed while connected to a valve by using a sealing torch. Care must be taken not to burn the valve. An insulation material placed between the valve and the torch is recommended.

Defrosting

After the freeze dry process is complete the ice that has accumulated on the collector coil must be removed. The Triad Freeze Dry System is equipped with a rapid defrost feature.

The following procedure should be followed when defrosting the collector coil:

1. Press the DEFROST button on the control panel. The defrost indicator above that button will illuminate. Allow unit to operate in this condition until all condensate is defrosted from the collector coil. Turn the system off by pressing RUN/STOP button or it will automatically shut off after 3 hours.
2. Place the drain hose in a suitable container to collect the condensation. Remove the drain plug from the drain hose.
3. Replace drain plug and remove drain pan to dispose of the ice and wipe dry.
4. Press the DEFROST button, the indicator will be off.
5. Wipe any excess liquid from inside of vacuum chamber, replace drain pan.



CAUTION

Utilization of acid requires immediate cleaning and neutralization after defrost or physical damage to the collector chamber and coil will result.

Do not attempt to chip ice from the collector coil as damage may occur.

Never attempt to start the vacuum pump when there is liquid in the collector chamber. This could result in damage to the vacuum pump.

Alarms

A number of events may occur during a lyophilization procedure that can adversely effect the operation of the Triad Freeze Dry System. If an event occurs, the alarm indicator will flash and beep.

The beep will automatically mute itself after one minute. The specific alarm can be identified by observing the display. The following “out of specification” conditions will initiate an alarm:

Shelf Temperature Variations

Once the shelf temperature has stabilized for 20 minutes, if the manual set point temperature or automatic hold temperature varies more than $\pm 2^{\circ}\text{C}$ as measured by the shelf temperature sensor, the Alarm Indicator and the word “SHLF” on the display will flash until the end of the run.

Collector Temperature Variations

If the temperature raises above -40°C and stays there for more than 20 minutes the Alarm Indicator and the word “COL” on the display will flash until the end of the run.

Vacuum

Once the system vacuum is low and stabilized at a point where it changes less than 0.020 mBar in 5 minutes, if the vacuum changes more than 0.500 mBar, the Alarm Indicator and the word “VAC” on the display will flash until the end of the run.

Vacuum Pump Service

When the vacuum pump service hours reaches 1000 hours. The Alarm Indicator and the VAC Indicator will flash on for 3 seconds and off for 1 second. When the vacuum is turned off the VAC Indicator will flash 1 second on and 3 seconds off. Resetting the vacuum pump service hours will cancel the alarm.

Shelf Temperature Set Point

If during a Ramp mode the system temperature stabilizes without reaching the set point temperature, the control will enter the next Hold mode. The Alarm Indicator will flash and the program indicator “Px” on the display will flash until the end of the run.

Power Failure

If a power failure occurs while a run is in progress, the Alarm Indicator and RUN/STOP Indicator will flash when the power is restored. Once power is restored, the process will continue as programmed if the collector temperature has not risen above -30°C . Pressing Run/Stop cancels the flashing warning.

Platen Tilt

If the stoppering platen tilts more than 4° while stoppering, the platen will stop. The display will read “SHELF UNEVENLY LOADED.”

Chapter 5:

Maintaining Your Freeze Dry System

Under normal operation, the freeze dry system requires little maintenance. However, the following maintenance schedule is recommended:

As needed:

1. The user has the responsibility for carrying out appropriate decontamination if hazardous material is spilled on or inside the equipment. This may be done by wiping the contaminated surfaces with a soft cloth dampened with alcohol. Alcohol may craze the acrylic door. Before using any cleaning or decontamination method except those recommended by Labconco, users should check with Labconco to determine that the proposed method will not damage the equipment.
2. Clean up all spills; remove liquids from the chamber.
3. Clean door and gasket using soft cloth, sponge or chamois and a mild, non-abrasive soap or detergent.
4. Check oil level of the vacuum pump. It should be between MIN and MAX. If the oil level is less than an inch (25.4 mm) above MIN, add oil to proper level.
5. If oil shows cloudiness, particles or discoloration, drain the pump and replace with fresh oil.
6. The use of acids requires immediate cleaning and neutralization after a run or physical damage will result.
7. When freeze drying biological substances, it may be necessary to decontaminate the system. A surface decontaminant should be used to clean the accessible surfaces.



CAUTION

The use of ethylene oxide is not recommended because of its hazardous and corrosive nature. Contact Labconco for additional information.

Monthly:

1. The rubber components on the freeze dry system may eventually deteriorate and require replacement. The effective life of rubber parts depends upon both their usage and the surrounding environment. Check all rubber hoses and gaskets and replace any that show signs of hardening, permanent set or deterioration.
2. Using a soft cloth, sponge or chamois and a mild, non-abrasive soap or detergent, clean the acrylic door.
3. Using a soft cloth, sponge, or chamois and a mild, non-abrasive soap or detergent, clean the exterior surfaces of the cabinet. Liquid spray cleaners and polishes may be used on the exterior surfaces. Do not use solvents to remove stains from the exterior surfaces as they may damage the finish.

Annually:

1. Every 12 months, or more often if the freeze dry system is operated in a dusty environment, the refrigeration system condenser should be cleaned. Using a vacuum cleaner with brush attachment, clean the condenser to ensure proper airflow for peak performance.

Chapter 6:

Using the RS232 Receptacle

The operation of the Triad Freeze Dry System can be monitored using a computer connected to the RS232 receptacle located on the rear panel of the freeze dryer. The computer cannot control the operation of the freeze dry system.

Computer Connection for Computer Interface

Check your computer to see which type of serial port is provided. Use one of the two connecting cables listed below:

1. Computers with a 25-pin D-sub male serial connector should use Connect Cable, Labconco part number 7537801, to connect a computer to the Labconco Freeze Dry System.
2. Computers with a 9-pin D-sub male serial connector should use Connect Cable, Labconco part number 7537800, to connect a computer to the Labconco Freeze Dry System.

The purpose of the RS232 interface is to send data to a data logging computer to monitor the state and activity of the freeze dry system. This data is half duplex data. The data properties are as follows:

1. Data Rate 2400 Baud
2. 8 bit word length
3. 1 Start bit, 1 Stop bit
4. No parity is transmitted
5. Standard ASCII character set

The time between data transmissions may be varied by the user to occur at 10, 30, 60, 300 or 600 second intervals. Press the display button until the set up indicator lights. Press the enter button until the RS232 screen appears on the display.

RS-232 TRANSMISSION RATE	
10	SECONDS

Press \uparrow or \downarrow until the desired time interval is shown. Press ENTER.

The format of the transmitted message with typical values is as follows:

PROG = 1 SEG = 2R TRM = 17 COL = -85 SHLF = -55 SAMP = -55 VAC = 180

Where:

PROG = Program
1 = Program 1 is selected
M = Manual Mode
S = Stop
SEG = Segment
2 = Segment 2 is operating
R = Ramping
H = Hold
E = End
P = Prefreeze
TRM = Time remaining of current hold segment in hours
COL = Collector
-85 = Collector temperature in °C
SHLF = Shelf
-55 = Shelf temperature in °C
SAMP = Sample Probe
-55 = Sample temperature in °C
VAC = Vacuum
180 = Vacuum in microbar
999 = Function not active

There are several commercially available software packages, which can read RS232 data and enter the data into a computer program such as a word processor (to create a text file) or spreadsheet (to tabulate and plot the data). Consult your laboratory supply dealer regarding the latest software available.

The following describes how to use an IBM compatible computer with Hyper Terminal™ software (included with Windows® 95, 98, XP or 2000 operating systems) to collect the RS232 data from your freeze dry system:

1. Make sure that the freeze dry system is properly connected to the communication port on the computer.
2. Open Hyper Terminal™ software.
 - i) Windows® 95 or 98 use:
START/PROGRAMS/ACCESSORIES.
 - ii) Windows® 2000 or XP use:
START/PROGRAMS/ACCESSORIES/COMMUNICATIONS

The first time Hyper Terminal™ is opened a dialogue box requesting an area code and phone number will appear. Enter the appropriate numbers and continue.

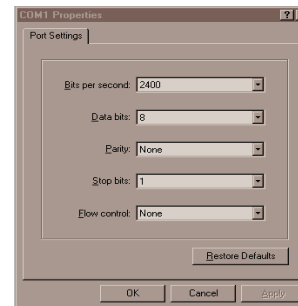
3. The “Connection Description” dialogue box will open. Type in a user defined name and select an icon for the new connection. Press “OK.”



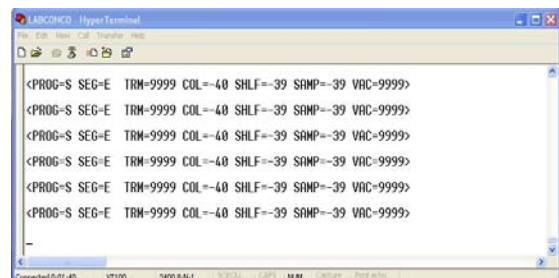
4. The “Connect To” dialogue box will open. Using the down arrow selection button, select the communication port to which the cable has been connected. Press “OK.”



5. The “Com X Properties” dialogue box will open. Enter the appropriate data properties and press “OK.”



6. When the freeze dry system main power switch is on, the data will be transmitted and updated at the time intervals selected by the user.



Chapter 7: Troubleshooting

Refer to the following if your freeze dry system fails to operate properly. If the suggested corrective actions do not solve your problem, contact Labconco for additional assistance.

Vacuum System

FreeZone[®] Triad[™] Freeze Dry Systems that are clean and dry and without samples attached should reach a vacuum of 0.133 mBar within 30 minutes and should achieve an ultimate vacuum of 0.040 mBar within 18 hours when the refrigeration is operating. If the freeze dry system does not obtain a satisfactory vacuum, perform the following maintenance tests.

Vacuum Pump

First make sure that the vacuum pump operates. If it fails to operate, check the electrical connections of the freeze dry system to the power source and then check the electrical connection of the vacuum pump to the freeze dry system. If the vacuum pump has a power switch, make sure that it is turned on. If the vacuum is not adequate when the vacuum pump is operating, proceed with the following steps:

1. Check the oil and ensure it is clear and clean. If the oil looks cloudy or has any particulates, replace the pump oil. If the oil is excessively dirty it may be necessary to flush the pump with clean oil several times. To flush the pump, run the pump 5 to 10 minutes to allow the oil to warm up. Drain the oil and refill with clean oil. Repeat as necessary.
2. Check the oil level in the pump. Ensure it is filled to the correct level.
3. Check vacuum hose connections from the pump to the freeze dry system and try running the unit.

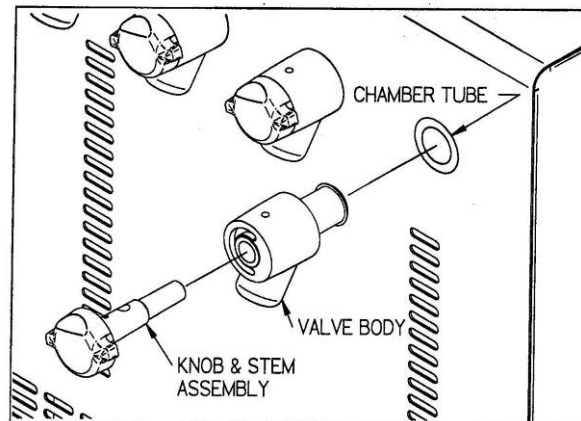
If vacuum problems continue, consider obtaining a second vacuum gauge capable of reading a vacuum of 0.010 mBar. It is often useful in determining if the vacuum pump is operating properly and the vacuum sensor reading is accurate.

4. Isolate the pump by disconnecting the vacuum hose from the freeze dry system. Deadhead the pump by inserting the vacuum sensor from a secondary vacuum gauge into the end of the vacuum hose and observe the vacuum reading obtained. Confirm that the pump is capable of achieving an ultimate vacuum less than 0.010 mBar or approximately 10 microns. If an inadequate vacuum reading is obtained, the pump has most likely failed and may need to be replaced or rebuilt.

Gaskets, Tubing, Connections and Sample Valves

1. Check all sample valves on the side of the cabinet and ensure all valves are closed or in the vent position.
2. Check all vacuum tubing for signs of deterioration or cracking.
3. Check all connections and make sure they are secure and leak tight.
4. Check the freeze dry system door gasket for indentations cracks or tears. Clean gaskets using a soft, lint free cloth or paper towel.
5. The freeze dry system chamber and collector must be dry.
6. When checking the sample valves, pull as much vacuum as possible. If a vacuum indication is displayed, wiggle or rotate the valves and watch the gauge for any fluctuation. Fluctuations can show a potential vacuum leak. If the valve seems to be in good condition, remove the valve and apply a thin coat of vacuum grease to the stem and the outside sealing surface of the valve body, and reinstall the valve. If the valve still seems to be the source of the problem, remove the valve and stopper the hole with a rubber stopper. Continue checking the other valves.

The illustration below shows how the sample valve installs on the chamber.

Figure 5

Diagnostic Mode

In the event that there is a problem with the Triad Freeze Dry System, a diagnostic mode is available. This mode can be used to diagnose problems by isolating individual system components to confirm their operation. With the unit Power Switch OFF, press the ⬇ button and turn the Power Switch ON. The display will show:

DIAGNOSTIC MODE

Press ⬆.

The display will show:

LEXSOL PUMP ON

The Lexsol pump will turn on or press ⬆.

The display will show:

LEXSOL AND VAC. PUMP ON

The Lexsol pump, vacuum pump and vacuum valve will turn on or press ⬆.

The display will show:

REFRIGERATION -- SHELF

The shelf valve opens and refrigeration system turns on or press \hat{u} .

The display will show:

REFRIGERATION -- COLL

The collector valve opens. The (shelf valve closes) refrigeration system remains on or press \hat{u} .

The display will show:

REF + LEX PUMP

The refrigeration system remains on. The Lexasol pump turns on or press \hat{u} .

The display will show:

DEFROST

The defrost valve opens or press \hat{u} .

The display will show:

VACUUM PUMP

The refrigeration system turns off. The vacuum pump turns on or press \hat{u} .

The display will show:

VAC VENT VALVE

The vacuum pump turns off. The vacuum control valve closes or press \hat{u} .

The display will show:

SHELF TILT VALVE

The vacuum control valve opens. The shelf tilt valve turns on (open) or press \hat{u} .

The display will show:

HEATER ON

The heater turns on for 3 minutes or press \hat{u} .

The display will go to monitor screen.

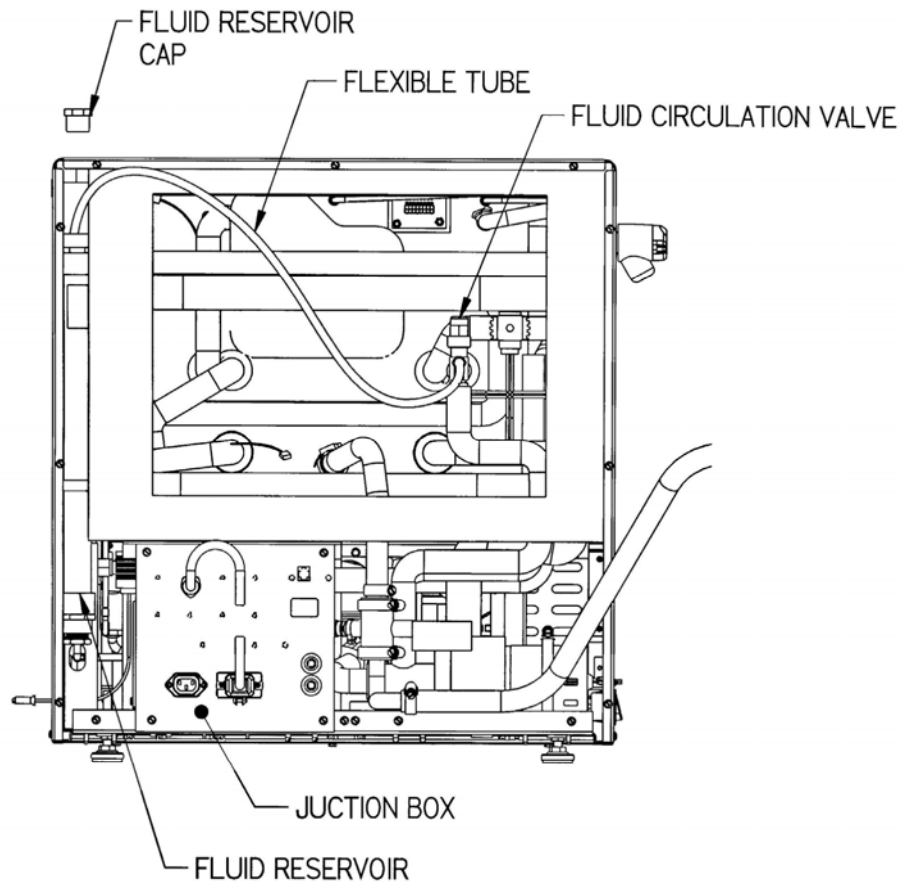
Refrigeration Module Operation

In the Pre-freeze mode under a no-load condition and an ambient temperature of 20°C, the freeze dry system refrigeration system can achieve a shelf temperature of -75°C or lower within 4 hours.

If any repairs are required on the refrigeration module, please call Labconco. Repairs should only be undertaken by a competent refrigeration technician or through an authorized Labconco service agency.

Filling the Fluid System

Figure 6



- (1) Disconnect power cord from supply outlet.
- (2) Make sure the Power Switch is in the OFF position.
- (3) Remove the rear cover to expose the marked back foam insulation. Cut through the foam along the marked lines and remove center cutout.
- (4) Remove the insulation and both caps from the fluid circulation valve.
- (5) Attach a flexible tube to the exposed port (service port). The port is 3/8" x 45° male flare.
- (6) Remove the fluid reservoir cap and place the opposite end of the tubing into the fluid reservoir.
- (7) Fill the reservoir slowly allowing the fluid to drain through the system. When the system appears full, prepare to start the circulation pump.
- (8) Open the fluid circulation valve by completely “down-seating” (turn top valve stem completely clockwise) to direct fluid to by-pass through tubing to fluid reservoir.
- (9) Connect the unit to the proper power source. Activate the pump by moving the Power Switch to the ON position, while holding down the ⏏ button. To start the diagnostic display, press the ⏏ button. The fluid system will circulate with the fluid going through the add-on tubing, purging itself of air. Add fluid when needed to keep fluid in the reservoir. After the fluid stream is established, tilt the entire freeze dry system up and down. Observe the fluid stream and repeat the tilt procedure if it is not free of bubbles. Fill reservoir to level indicated by the label.
- (10) Turn power switch to the OFF position and disconnect power cord from supply outlet.
- (11) Close “up-seat” fluid circulation valve (turn valve stem completely counter-clockwise, closing the fluid/tubing by-pass).
- (12) Remove the tubing. Replace the caps on the valve and replace insulation and covers. Replace the cap on the tank.
- (13) Replace insulation and seal air tight. Replace rear cover.

Heat Transfer Fluid Precautions

The fluid's name and manufacturer are:

Name: Lexol 542
Manufacturer: Santa Barbara Chemical Co.
927 Indio Muerto
Santa Barbara, CA 93140
805-963-7793



WARNING

The fluid used for heat transfer in this system is combustible and hazardous. Leaks and spills should be attended to immediately or serious injury could occur.

Handling and Storage Precautions – Dirty Solvent: Store in accordance with all applicable regulations. Tighten caps and store in a cool area.

Precautions if Material is Released or Spilled: Spills should be contained immediately. Spills may be soaked up with absorbent materials, placed in closed containers, labeled, stored and disposed of properly. Persons performing this work should wear adequate personal protective equipment and clothing.

Disposal of Non-Recyclable Solvents: Dispose of in accordance with all federal, state, and local health and pollution regulations. Follow same guidelines as when disposing kerosene.

Heat Transfer Fluid First Aid Procedures

Eye Contact: Immediately flush eyes with fresh water for at least 15 minutes. If irritation persists, get medical attention.

Skin Contact: Wash contaminated areas with soap and water. Remove contaminated clothing and footwear. Wash clothing before reuse. Discard footwear which cannot be decontaminated. Medical attention may be required.

Inhalation: Remove patient to fresh air. If breathing stops, give artificial respiration. Get medical attention immediately, if required.

Ingestion: Get medical attention immediately. Do not induce vomiting.

Chapter 8:

Modifying Your Freeze Dry System Display

Your Triad Freeze Dry System has been carefully calibrated and tested before shipping, however under certain circumstances it may be necessary to adjust the calibration of the vacuum display or to reset factory settings. These adjustments may be necessary if the freeze dry system requires service.

An offset may be added to the vacuum display. Use an accurate traceable reference to compare the readings.

Vacuum offset may be varied ± 0.010 mBar in 0.001 mBar increments.

Modifying the Display

To enter an offset or to restore all factory settings, perform the following steps:

- Press the DISPLAY button until the SET UP INDICATOR is lit.
- Press and hold DISPLAY button for 10 seconds.

The display will first show the screen used for setting Auto mode parameters and then will show:

PRODUCTION MENU
MASTER RESET?

If you want to restore all factory settings hold ENTER approximately 5 seconds.

This will reset:

Vacuum units to mBar
Vacuum offset to 0 mBar
Temperature units to °C
Refrigeration service hours to 0
Vacuum total hour to 0
Vacuum service hour to 0
Refrigeration total hours to 0
RS232 transmission rate to 10 seconds

At the completion of Master Reset, the display will return to the monitor mode.

If you do not want to restore all factory settings, but want to adjust the vacuum offset, press DISPLAY briefly and the display will show:

VACUUM OFFSET: 0 mBar

Press \uparrow or \downarrow until the appropriate offset is shown.
Each number represents an offset of 0.001 mBar

Press DISPLAY and the display will show:

COLLECTOR TEMP. OFFSET: 0°C

Press \uparrow or \downarrow until the appropriate offset is shown.
Each number represents an offset of 1°C.

Press DISPLAY and the display will show:

SHELF TEMP. OFFSET: 0°C

This is the probe mounted in the shelf. Temperature from this probe is displayed during Pre Freeze.

Press \uparrow or \downarrow until the appropriate offset is shown.
Each number represents an offset of 1°C.

Press DISPLAY and the display will show:

LEXSOL TEMP. OFFSET: 0°C

This is the probe mounted to the Lexsol tube in the back. Temperature from this probe is displayed as “SHLF TEMP” during sequences other than PRE FREEZE.

Press \uparrow or \downarrow until the appropriate offset is shown. Each number represents an offset of 1°C.

Press DISPLAY and the display will show:

SAMPLE TEMP. OFFSET: 0°C

Press \uparrow or \downarrow until the appropriate offset is shown.
Each number represents an offset of 1°C.

Press DISPLAY and the display will show:

PRODUCTION MENU: MASTER RESET?

Wait approximately 10 seconds and the display will default to show operational parameters.

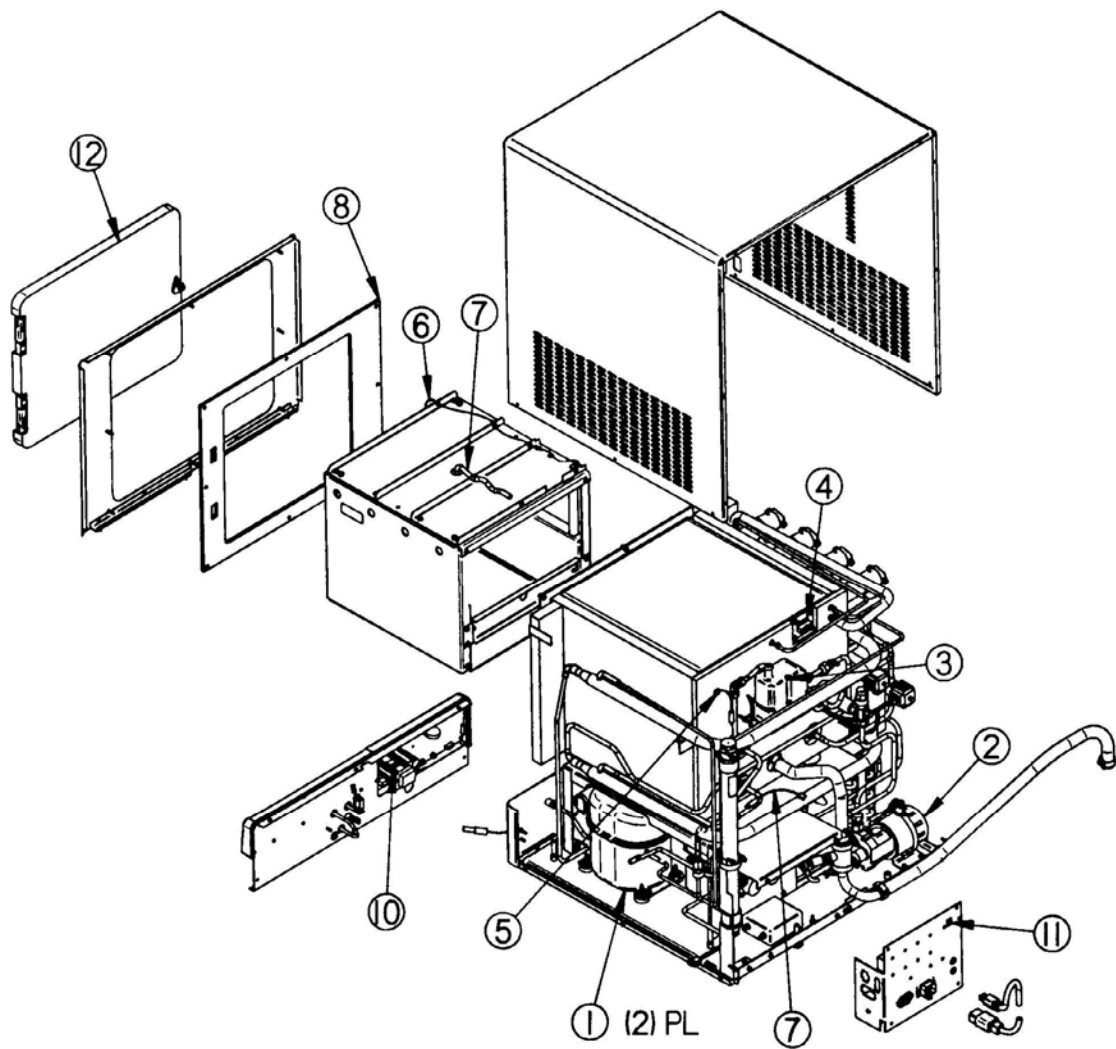
Appendix A:

Freeze Dry System Components

The following pages list components that are available for your freeze dry system. The parts shown are the most common replacement parts. If other parts are required, contact Product Service.

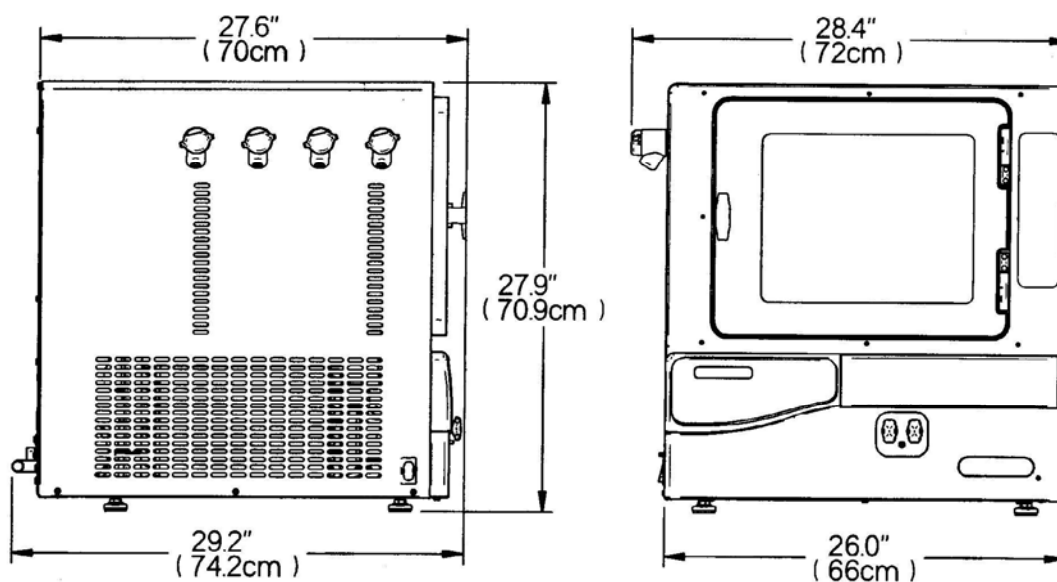
Item	Part No.	Qty	Description
1	7591801	2	Compressor 230V – 50 Hz
	7591802	2	Compressor 230V – 60 Hz
2	7501902	1	Lexsol Pump 230V
3	7510901	1	Heater 230V
4	7754800	1	Pass thru Board
5	7515300	2	Temperature Sensor – Lexsol Collector
6	7372500	2	Temperature Sensor – Sample/Shelf
7	7765501	1	Diaphragm
8	7424100	1	Door Gasket
9	7766801	1 Gal.	Heat Transfer Fluid (Not Shown)
10	7395300	1	Printed Circuit Board Control
11	7448000	1	Printed Circuit Board RS232
12	7425500	1	Door Assembly

Figure 7



Appendix B: Freeze Dry System Dimensions

Figure 8



Serum Bottle Capacity of the Triad Freeze Dry System

Size	Labconco No (100 pcs.).	Shelf Capacity
2 ml	7575010	391
3 ml	7575210	441
5 ml	7573010	233
10 ml	7573210	196
20 ml	7573410	121
30 ml	7573610	86
50 ml	7573810	64
100 ml	7574010	42
125 ml	7574210	36

Appendix C:

Freeze Dry System Specifications

This Appendix contains technical information about the freeze dry system's electrical specifications and environmental operating conditions.

Electrical Specifications

Catalog Number	Description	Voltage		Frequency	Phase	Amperage
		Nominal	Operating Range			
7400030	Triad Freeze Dry System	230	198-254	50	1	12
7400040	Triad Freeze Dry System	230	187-253	60	1	12

Environmental Conditions

- Indoor use only.
- Maximum altitude: 6562 feet (2000 meters).
- Ambient temperature range: 41° to 104°F (5° to 40°C).
- Maximum relative humidity: 80% for temperatures up to 88°F (31°C), decreasing linearly to 50% relative humidity at 104°F (40°C).
- Transient over voltages according to Installation Categories II (Over voltage Categories per IEC 1010). Temporary voltage spikes on the AC input line that may be as high as 1500V for 115V models and 2500V for 230V models are allowed.
- Used in an environment of Pollution degrees 2 (i.e., where normally only non-conductive atmospheres are present). Occasionally, however, a temporary conductivity caused by condensation must be expected, in accordance with IEC 664.

Performance Specifications

Collector Minimum Temperature (°C)	-85
Collector Capacity in 24 Hours (Liters)	1.84
Collector Total Capacity (Liters)	2.5
Shelf Minimum Temperature (°C)	-75
Shelf Control Temperature Range (°C)	-55 to +50
Shelf Size (Inches)	12.4 x 14.5 x 5.75 high
Number of Shelves	1
Defrost Method	Hot Gas
Time to Defrost 2.0L (Hr)	1.5
All Temperature Specification @ 20°C Ambient	

Appendix D: Freeze Dry System Accessories

The following accessories are available for the Triad Freeze Dry System.

PART #	DESCRIPTION
7537800	Cable Connect the RS232 output from the Triad Freeze Dry System to an IBM compatible computer with a 9-pin serial data port.
7537801	Cable Connects the RS232 output from the Triad Freeze Dry System to an IBM compatible computer with a 25-pin serial data port.
7756100	Tray with Slide-Out Bottom 14" wide x 12" deep. Stainless steel tray has separate bottom, which slides out allowing serum bottles, and ampules direct contact with the shelf to facilitate the drying process.
7756200	Bulk Tray 15" wide x 11-1/2" deep. Stainless steel tray will contain liquids for bulk drying.
7516200	Support Grid 7" wide x 7" deep. Stainless steel tray with removable plastic grid provides support for stoppering ampules and other small specimen containers while stoppering under vacuum. Grid holds 144 ampules of 12 mm diameter.
7739403	Vacuum Pump Two stage direct drive pump, 195 liters/minute. 230 VAC, 50/60 Hz, single phase, 4.0 amps. Includes Pump Exhaust Filter/#1473400.
7439200	Vacuum Pump Two stage direct drive pump, 173 liters/minute. 230 VAC, 50/60 Hz, single phase, 2.9 amps. Includes Pump Exhaust Filter/#7670400.
7769800	Vacuum Pump Corrosion Resistant Two stage direct drive pump, 173 liters/minute. 230 VAC, 50/60 Hz, single phase, 2.9 amps. Includes Pump Exhaust Filter/#7670400.

PART #	DESCRIPTION
1472200	Pump Inlet Filter Disposable filter that prevents oil back streaming and protects vacuum pump from sub micron particles. Fits Vacuum Pump #7739403.
1473400	Pump Exhaust Filter Disposable filter that removes visible oil mist and odor from vacuum pump exhaust. Fits Vacuum Pump #7739403.
1473200	Replacement Element, Oil Mist, Pump Exhaust Filter Fits Pump Exhaust Filter #1473400.
1473300	Replacement Element, Odor, Pump Exhaust Filter, package of 5 Fits Pump Exhaust Filter #1473400 or Vacuum Pump #7739403.
7670400	Replacement Filter – Element Disposable filter that removes oil mist from the vacuum pump exhaust. Fits Vacuum Pumps #7439200 and #7769800.
1988000	Vacuum Pump Oil, 1 Liter A molecularly distilled hydrocarbon oil with low vapor pressure. For Vacuum Pump #7739403.
7772700	Vacuum Pump Oil 1 Liter A mineral oil with low vapor pressure. For Vacuum Pumps #7439200 and #7769800.
7772000	Soda Acid Trap Secondary trap that prevents migration of corrosive chemicals into vacuum pump.
7772100	Replacement Media for Soda Acid Trap
7772500	Carbon Solvent Trap Secondary trap that prevents migration of organic solvents into vacuum pump.
7772600	Replacement Activated Carbon Media for Carbon Solvent Trap
7509600	Sample Valve Kit Includes neoprene valve body, knob and installation parts.

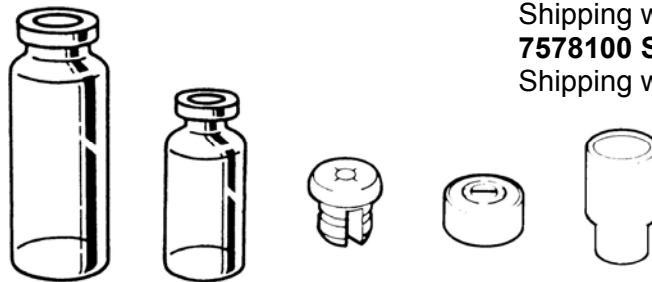
Serum Bottles and Vials



Perfect for long term storage of freeze dried samples. Labconco Serum Bottles and Treaded Vials are specifically designed for lyophilization applications. Their uniform thin wall construction ensures even freezing and drying. Bottles and vials are ideal containers for use in the FreeZone Stoppering Tray Dryer. Serum bottles also connect to valve ports on drying chambers and manifolds.

Serum Bottles

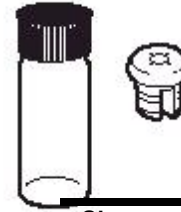
Serum Bottles, Stoppers and seals are supplied in packages of 100.



Size	20 mm Corkage	13 mm Corkage	Split Stoppers	Aluminum Seals	Sleeve-Type Stoppers
2 ml		7575010	7576010	7577010	
3 ml		7575210	7576010	7577010	
5 ml	7573010		7576210	7577110	757710
10 ml	7573210		7576210	7577110	7577510
20 ml	7573410		7576210	7577110	7577510
30 ml	7573610		7576210	7577110	7577510
50 ml	7573810		7576210	7577110	7577510
100 ml	7574010		7576210	7577110	7577510
125 ml	7574210		7576210	7577110	7577510

Threaded Vials

Stoppers and threaded vials with Screw Caps are supplied in packages of 200.



Size	Vials with Screw Caps	Stoppers
5 ml	7762300	7762200
10 ml	7762600	7762200



Seal Crimper

Secures tear-away Aluminum Seals.

7578000 Seal Crimper for 13 mm corkage.

Shipping weight 3 lbs. (1.4 kg)

7578100 Seal Crimper for 20 mm corkage.

Shipping weight 3 lbs. (1.4 kg)

Fast-Freeze® Flasks



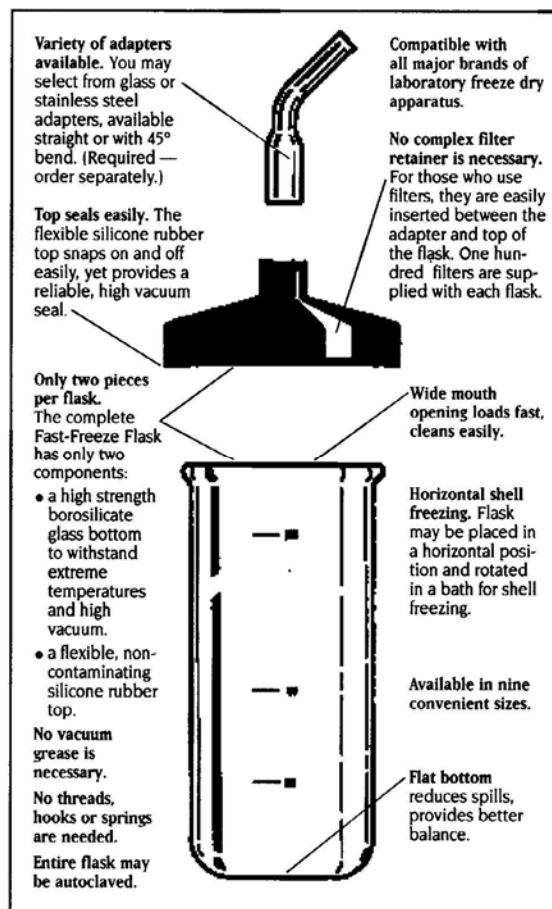
Fast-Freeze Flasks are specially designed to be easier to handle, faster to load and more convenient to use than other freeze dry glassware now in your laboratory. Compatible with all major brands of laboratory freeze dry equipment, Fast-Freeze Flasks eliminate the risk of contamination from vacuum grease, reduce spillage of valuable samples and require no washers, gaskets or retainers.

How to select Fast-Freeze Flasks for your Freeze Dry System

Select fast-Freeze Flasks based on your sample sizes. Flasks should be filled no more than one-third of their volume so that maximum surface area is achieved and efficient lyophilization is assured. A complete Fast-Freeze Flask includes a rubber top, glass bottom and a supply of filter paper. Tops, bottoms and filter paper are available separately as replacement components. Adapters are required to attach flasks to freeze dry valve ports.

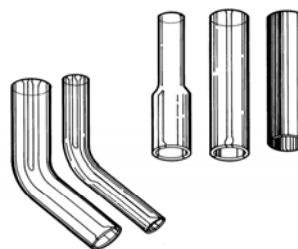
Flask Size	Complete Flask	Flask Bottom	Flask Top	Flask Top Adapter Diameter*	Dimensions/Flask Bottom H x D
40 ml	7540000	7542000	7544000	1/2"	76mm x 34mm
80 ml	7540200	7542200	7544000	1/2"	115mm x 34mm
120 ml	7540300	7542300	7544200	3/4"	68mm x 59.2mm
150 ml	7540400	7542400	7544200	3/4"	85mm x 59.2mm
300 ml	7540600	7542600	7544200	3/4"	145mm x 59.2mm
600 ml	7540800	7542800	7544400	3/4"	135mm x 90.2mm
900 ml	7540900	7542900	7544400	3/4"	190mm x 90.2mm
1200 ml	7541000	7543000	7544400	3/4"	240mm x 90.2mm
2000 ml	7541200	7543200	7544400	3/4"	380mm x 90.2mm

*Valves on the freeze dry system, drying chambers and manifolds offered in this catalog fit adapters of both 1/2" and 3/4" sizes. Size information is provided for selecting adapters for older Labconco models and other freeze dryers.



Adapters

Add the adapters for connecting the Fast-Freeze Flasks to the valve ports on your drying chamber or manifold. Choose borosilicate glass or stainless steel adapters in 1/2" and 3/4" diameters



7544810 Replacement Filter Paper
1000 (10 packages of 100). 1" diameter (28mm). Pore size 17 micron. Shipping weight 0.5lb.

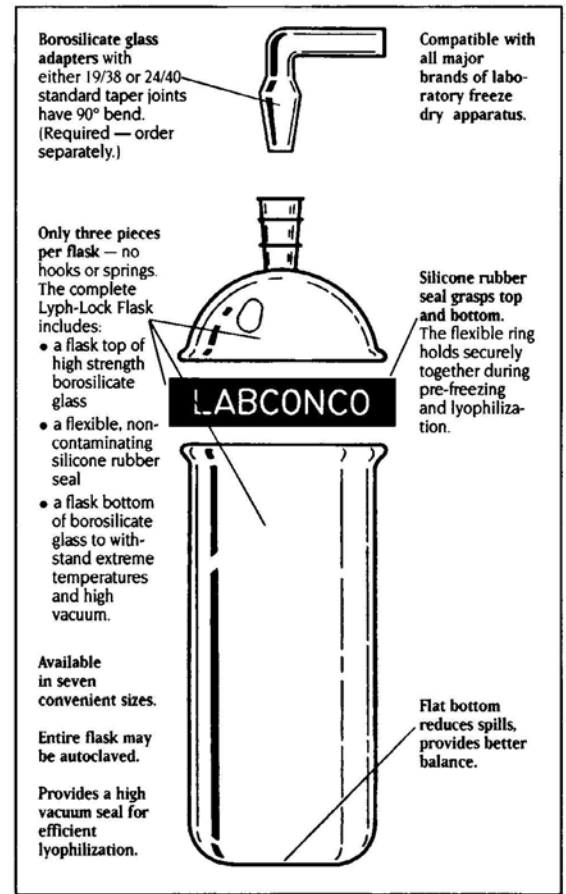
Lyph-Lock® Flasks



Lyph-Lock Flasks simplify your Lyophilization procedures because they have only three pieces per flask – a high strength borosilicate glass top and bottom with a silicone rubber ring seal. The unique wide-mouth design helps you load samples easily and lyophilize efficiently.

How to select Lyph-Lock Flasks for your Freeze Dry System

Select Lyph-Lock Flasks fast- based on your sample sizes. Flasks should be filled no more than one-third of their volume so that maximum surface area is achieved and efficient lyophilization is assured. A complete Lyph-Lock Flask includes a glass top and bottom and a rubber ring seal. Tops, bottoms and seals are available separately as replacement components. Adapters are required to attach flasks to freeze dry valve ports.



Adapters

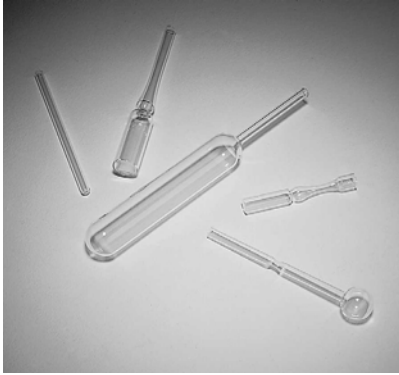
Add the adapters for connecting the Lyph-Lock Flasks to the valve ports on your drying chamber or manifold.

90° Bend Adapter	Description
7568000	Connects 19/38 STj Flask Top to ½" valve*
7568200	Connects 19/38 STj Flask Top to ¾" valve*
7568400	Connects 24/40 STj Flask Top to ½" valve*
7568600	Connects 24/40 STj Flask Top to ¾" valve*

Flask Size	Complete Flask 19/38 STj	Complete Flask 24/40 STj	Flask Top 19/38 STj	Flask Top 24/40 STj	Lyph-Lock Seal	Flask Bottom	Dimensions/ Flask Bottom H x ID
25 ml	7550000	7550000	7552000	7556000	7559000	7557000	37mm x 34mm
50 ml	7550200	7554200	7552000	7556000	7559000	7557200	67mm x 34mm
100 ml	7550400	7554400	7552200	7556200	7559200	7557400	50mm x 59.2mm
250 ml	7550600	7554600	7552200	7556200	7559200	7557600	110mm x 59.2mm
500 ml	7550800	7554800	7552400	7556400	7559400	7557800	103mm x 90.2mm
750 ml	7550900	7554900	7552400	7556400	7559400	7557900	145mm x 90.2mm
1000 ml	7551000	7555000	7552400	7556400	7559400	7558000	187mm x 90.2mm

*Valves on the freeze dry system, drying chambers and manifolds offered in this catalog fit adapters of both ½" and ¾" sizes. Size information is provided for selecting adapters for older Labconco models and other freeze dryers.






Ampules



Labconco Ampules are fabricated of highest quality borosilicate glass for strength and durability. They are available in five different configurations to provide the ideal ampule for your specific lyophilization requirements, from tray drying to flame sealing applications.

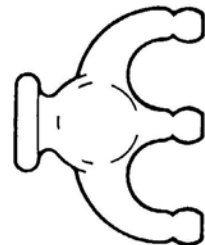
Ampules

Ampules are supplied in packages of 100.

					
Size	Straight	Bulb-Type	Pre-Scored Flat Bottom	Round Bottom	Stoppering
1 ml	7572710	7570010	757010		
2 ml			7570810		7572310
4 ml	757210				
5 ml		7570210	7571010		
10 ml			757110	7571610	
25 ml				7571810	
50 ml				7572010	

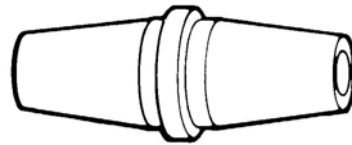
Trident Adapter

7762700 Three Way Adapter permits attachment of three ampules to a single valve port. Ampules are attached using Ampule Valve Adapters or 1/8" surgical tubing (not provided). Cavity in adapter body can be filled with cotton fiber media to help prevent contamination between samples. Shipping weight 3 oz. (0.1 kg).



Oxygen/Natural Gas Sealing Torch

7578500 Torch specifically designed for flame sealing freeze dry ampules. Seals all types of heat-resistant glass. Connects to natural gas, butane or propane and oxygen with 1/4" ID hose connectors. Shipping weight 3 lbs. (1.4 kg).



Ampule Valve Adapters

7593401 Adapter connects ampules to valve ports or stainless steel stems. Ten per package. Shipping weight 0.5 lbs.



Stoppers

7572510 Stopper for Stoppering Ampules 7572310. 100 per package. Shipping weight 3 lbs. (1.4 kg).

DECLARATION OF CONFORMITY

Application Council Directive(s): 73/23/EEC, 89/336/EEC, 2002/95/EC (ROHS),
2002/96/EC (WEEE)

Standard(s) to which conformity is declared: EN61010-1, EN55014, EN55104,
EN55022, EN61000-3-2, EN61000-3-3

Manufacturer's Name: Labconco Corporation

Manufacturer's Address: 8811 Prospect Avenue
Kansas City, MO 64132 USA

Importer's Name: See Shipping/Customs Documents

Importer's Address: See Shipping/Customs Documents for your equipment

Type of Equipment: Laboratory Equipment – Sample Preparation

Model No.: Stoppering Tray Dryer – 230V - #7948030
Triad Freeze Dry/Tray Dryer - 230V - #7400030

Serial No.: Various – See Individual Declaration

Year of Manufacture: 2004 and Subsequent

I, the undersigned, hereby declare that the equipment specified above conforms to the
above Directive(s) and Standard(s).

See individual Declaration of Conformity which
will be signed by the importer for your country.

Place:

(Signature)

Date:

(Full Name)

(Position)

Labconco P/N 3696010, REV E, ECO E077