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CHAPTER 1
INTRODUCTION

Congratulations on your purchase of a Labconco Freeze Dry System. Models are available for operation on 115V or 230V.

The Labconco FreeZone® Plus 2.5 Liter Freeze Dry System is designed for laboratory lyophilization procedures. It 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

Dehydration is an important process 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 microorganisms). 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 is removed from frozen materials by converting the frozen water directly into its 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 water; the use of a vacuum pump to enhance the removal of water vapor from the surface of the sample; the transfer and deposit of water vapor onto a collector; and the removal of heat due to ice formation from the collector by means of a refrigeration system. In essence, the freeze dry process is a balance between the heat absorbed by the sample to vaporize the water 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 three 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 freeze dryer capacities.

Up to the point of overloading the system, the greater the surface area of the sample, the greater 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 flask 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. This is accomplished by the collector and the vacuum pump. The collector, which should be at least 10 to 15°C colder than the eutectic temperature (melting temperature) of the sample, traps vapor as ice. Since the vapor pressure at the collector is less 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 most applications, the maintenance of a vacuum of 133×10^{-3} mBar or less is required for freeze drying to occur.

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 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 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 there is a simple sugar such as a 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 to less than 133×10^{-3} mBar 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. If the eutectic temperature of the sample is –40 to –60°C, the freeze dry system selected for use must be equipped with

cascade type refrigeration so that the collector temperature can be cooled to below –75°C, or a dry ice/solvent trap can be used between the collector and the vacuum pump.

Samples Containing Volatile Substances

In certain cases the solvent in a sample to be freeze dried may contain volatile components such as acetic acid, formic acid or pyridine. In addition to these substances having an effect on the eutectic temperature, they may enhance 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, freeze drying 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.

About This Manual

This manual is designed to help you learn how to install, use, and maintain your Freeze Dryer. Instructions for performing routine maintenance and making minor modifications to your Freeze Dryer are also included.

Chapter 1: Introduction provides a brief overview of the Freeze Dryer, explains the organization of the manual, and defines the typographical conventions used in the manual.

Chapter 2: Prerequisites explains what you need to do to prepare your site before you install your Freeze Dryer. Electrical requirements are discussed.

Chapter 3: Getting Started contains the information you need to properly unpack, inspect and install your Freeze Dryer.

Chapter 4: Using Your Freeze Dryer discusses the basic operation of your Freeze Dryer. Information on how to attach samples and run the Freeze Dryer is included.

Chapter 5: Maintaining Your Freeze Dryer explains how to perform routine maintenance on your Freeze Dryer.

Chapter 6: Modifying Your Freeze Dryer describes how to attach a recorder or computer for monitoring the operation.

Chapter 7: Troubleshooting contains a table of problems you may encounter while using your Freeze Dryer, including the probable causes of the problems, and suggested corrective actions.

Appendix A: Freeze Dryer Components contains labeled diagrams of the components of the Freeze Dryer.

Appendix B: Freeze Dryer Dimensions contains comprehensive diagrams showing the dimensions for the Freeze Dryer.

Appendix C: Freeze Dryer Specifications contains product specifications. Wiring diagrams for both the 115V and 230V Freeze Dryer are also included.

Appendix D: Freeze Dryer Accessories lists the part numbers and descriptions of all of the accessories available for your Freeze Dryer.

Typographical Conventions

Recognizing the following typographical conventions will help you understand and use this manual:

- Book, chapter, and section titles are shown in italic type (e.g., *Chapter 3: Getting Started*).
- Steps required to perform a task are presented in a numbered format.
- Comments located in the margins provide suggestions, reminders, and references.
- Critical information is presented in boldface type in paragraphs that are preceded by the exclamation icon. Failure to comply with the information following an exclamation icon may result in injury to the user or permanent damage to your Freeze Dryer.

- Important information is presented in capitalized type in paragraphs that are preceded by the pointer icon. It is imperative that the information contained in these paragraphs be thoroughly read and understood by the user.

Your Next Step

If your Freeze Dryer needs to be installed, proceed to *Chapter 2: Prerequisites* to ensure your installation site meets all of the requirements. Then, go to *Chapter 3: Getting Started* for instructions on how to install your Freeze Dryer and make all of the necessary connections.

For information on the operational characteristics of your Freeze Dryer, go to *Chapter 4: Using Your Freeze Dryer.*

If your Freeze Dryer is installed and you need to perform routine maintenance on the Freeze Dryer, proceed to *Chapter 5: Maintaining Your Freeze Dryer.*

For information on making modifications to the configuration of your Freeze Dryer go to *Chapter 6: Modifying Your Freeze Dryer.*

Refer to *Chapter 7: Troubleshooting* if you are experiencing problems with your Freeze Dryer.

CHAPTER 2
PREREQUISITES

Before you install your Freeze Dryer, you need to prepare your site for installation. Carefully examine the location where you intend to install your Freeze Dryer. 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:

- the electrical supply requirements.
- the vacuum pump requirements.
- chamber or manifold requirements.

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

Electrical Requirements

The Freeze Dryer requires a dedicated electrical outlet. This outlet requires a 20 Amp circuit breaker or fuse for models rated at 115V (60 Hz). A 10 Amp circuit breaker or fuse is required for models rated at 230V (50 Hz). 115V models are equipped with a 20 Amp NEMA 5-20P plug. 230V models are not equipped with a plug. It will be necessary to install a plug to match the available receptacle.

Location Requirements

The Freeze Dryer 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 through the grill on the front panel and exhausts it out through the back and sides of the cabinet. A minimum of 3" must be allowed between the back and sides of the Freeze Dryer and adjacent wall surfaces. Restriction of airflow during operation could adversely affect performance.

Vacuum Pump Requirements

A vacuum pump must be provided by the user. A vacuum pump with a displacement of 113 liters per minute and 0.2 x 10^{-3} mBar blank off pressure is adequate for most samples. The inlet fitting on the vacuum pump must be suitable for 0.75 ID hose which is provided.

Vacuum pumps used with 115V models should be equipped with a 115V, 15 Amp NEMA 5-15P plug. Vacuum pumps used with 230V models should be equipped with a "reverse" IEC 320 plug. This will allow the vacuum pump to be plugged into the receptacle on the back of the Freeze Dryer. See *Appendix D: Freeze Dryer Accessories* for vacuum pumps available from Labconco.

Chamber or Manifold Requirements

A freeze drying chamber or manifold must be purchased separately. These allow samples to be attached to the Freeze Dryer. See *Appendix D: Freeze Dryer Accessories* for available chambers and manifolds.

Space Requirements

Refer to *Appendix C: Freeze Dryer Specifications* for dimensional drawings of the Freeze Dryer.

Your Next Step

After you have determined that the location for your Freeze Dryer accommodates the installation and operational requirements, you are ready to unpack and install your Freeze Dryer. Proceed to *Chapter 3: Getting Started.*

CHAPTER 3
GETTING STARTED

Now that the site for your Freeze Dryer is properly prepared, you are ready to unpack, inspect, install and test your Freeze Dryer. Read this chapter to learn how to:

- unpack and move your Freeze Dryer.
- set up your Freeze Dryer.
- connect the electrical supply source to your Freeze Dryer.
- properly exhaust your Freeze Dryer.
- safely use solvents with your Freeze Dryer.

The Freeze Dryer weighs over 135 lbs. (61 Kg). The carton allows for lifting with a mechanical lift truck or hand truck. If you must lift the Freeze Dryer manually, use at least two (2) persons and follow safe lifting guidelines.

Unpacking Your Freeze Dryer

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

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

DO NOT RETURN GOODS WITHOUT THE PRIOR AUTHORIZATION OF LABCONCO. UNAUTHORIZED RETURNS WILL NOT BE ACCEPTED.

IF YOUR FREEZE DRYER WAS DAMAGED IN TRANSIT, YOU MUST FILE A CLAIM DIRECTLY WITH THE FREIGHT CARRIER. LABCONCO CORPORATION AND ITS DEALERS ARE NOT RESPONSIBLE FOR SHIPPING DAMAGE.

Do not discard the carton or packing material for your Freeze Dryer until you have checked all of the components and installed and tested the Freeze Dryer.

Freeze Dryer Components

As previously mentioned, the Freeze Dryer System is available in 115V or 230V. Locate the model of Freeze Dryer you received in the following table. Verify that the components listed are present and undamaged.

Catalog #	Product Description
76700-00	Freeze Dryer – 115V, 60 Hz
76700-01	Freeze Dryer – 230V, 50 Hz

Plus the Following:

Part #	Component Description
74776-00	User's Manual
13364-00	Power Cord – 115V
	or
13373-00	Power Cord – 230V
76460-00	Tubing
19676-00	Clamp (2)

If you do not receive one or more of the components listed for your Freeze Dryer, or if any of the components are damaged, contact Labconco Corporation immediately for further instructions.

Setting Up Your Freeze Dryer

After you verify receipt of the proper components, move your Freeze Dryer to the location where you want to install it. Then, follow the steps listed below.

Component Orientation & Hose Connections

The refrigeration system in the Freeze Dryer exhausts air through the rear of the cabinet. A minimum of 3" should be allowed between the back of the Freeze Dryer 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.

Electrical Connection

Plug the power cord into the receptacle on the back of the Freeze Dryer and plug the other end into a suitable power receptacle. Plug the power cord from the vacuum pump into the receptacle on the back of the Freeze Dryer. If the vacuum pump has an off/on switch, turn the switch on. The vacuum pump will be controlled by the Freeze Dryer.

Drying Chamber Installation

The drying chamber, purchased separately, may be positioned directly above and centered over the collection chamber. When vacuum is applied to the system, the chamber will be held securely in place.

Drying Manifold Installation

If a drying manifold is to be installed, first place the accessory acrylic attachment port lid 74774-00 over the collection chamber. Then the manifold can be inserted into the hole on the lid making sure that the rubber gasket is between the lid and the flange on the manifold.

Solvent Safety Precautions

Solvents used in the Freeze Dryer may be flammable or hazardous. Use extreme caution and keep sources of ignition away from the solvents. When using flammable or hazardous solvents, the vacuum pump should be vented to or operated inside 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.

Several components of the Freeze Dryer, which are located inside the chamber, are made of stainless steel which can be attacked by acids. Use of acids can result in degradation of the product and the vacuum pump.

Use care when using aggressive liquids which can damage the Freeze Dryer and thoroughly clean the Freeze Dryer after each use. Instructions for cleaning are in *Chapter 5: Maintaining Your Freeze Dryer*.

WARNING: The disposal of substances used in connection with this equipment may be governed by various Federal, State or local regulations. 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.

Your Next Step

The installation and setup of your Freeze Dryer is now complete. To learn how to load and operate your Freeze Dryer, proceed to *Chapter 4: Using Your Freeze Dryer*. To make a modification to the configuration of your Freeze Dryer, proceed to *Chapter 6: Modifying your Freeze Dryer*. To perform additional diagnostics on your Freeze Dryer, proceed to *Chapter 7:Troubleshooting*. To learn about the maintenance requirements for your Freeze Dryer, proceed to *Chapter 5: Maintaining Your Freeze Dryer.*

CHAPTER 4
USING YOUR FREEZE DRYER

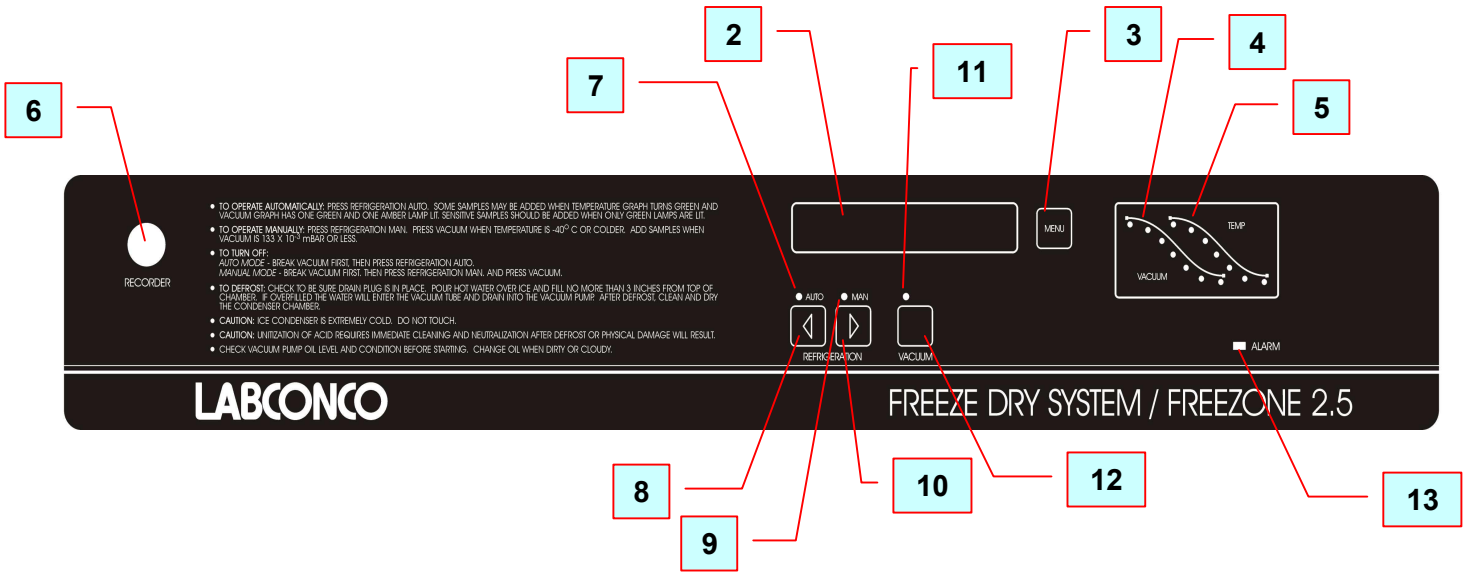
After your Freeze Dryer has been installed as detailed in *Chapter 3: Getting Started*, you are ready to begin using your Freeze Dryer. Read this chapter to learn how to:

- operate the controls.
- understand the display.
- connect samples.

Do not use the Freeze Dryer in a manner not specified by the manufacturer (refer to *Appendix C: Freeze Dryer Specifications*). The electrical protection properties of the Freeze Dryer may be impaired if the Freeze Dryer is used inappropriately.

Freeze Dryer Controls

The control panel for the Freeze Dryer is shown below with a description about its function.



1. Main Power Switch (Not Shown) – Turns the unit on or off. This switch is located on the right side of the unit near the front.
2. LCD Display – Displays system parameters and alarm messages. Parameters displayed are system vacuum (10^{-3} mBar) and collector temperature (°C). Displays type of alarm when an alarm has occurred.
3. Menu Switch – This switch is used to change the display from system parameters to alarm messages.
4. Vacuum Graph Display – This display indicates the relative system vacuum level. The highest LED indicates that the vacuum level is above 2000 x 10^{-3} mBar. The indicators will sequence down when the vacuum level reaches 2000, 1000, 800, 600, 450, 133 x 10^{-3} mBar. The lower green LED flashes when the system vacuum level is 450 to 133 x 10^{-3} mBar and illuminates steadily below 133 x 10^{-3} mBar.

5. Collector Temperature Graph Display – This display indicates the temperature of the collector. The highest LED indicates the collector temperature is warmer than 10°C. The indicators will sequence down when the temperature reaches 10, 0, -10, -20, -30 –40°C. When the collector temperature is –40°C or lower the green indicator will light.
6. Recorder Jack – This standard 8 pin DIN connector allows the user to monitor the system parameters with a remotely connected instrument. Signal outputs from this jack are 1) collector temperature, 2) vacuum level and 3) RS232 data stream.
7. Auto Mode Annunciator – When lit, the green LED indicates that the Freeze Dry unit is in the Auto Mode. In this mode, the vacuum pump will start when the collector temperature reaches –40°C.
8. Auto Mode Switch – Used to start or stop the refrigeration and the Auto Mode process.
9. Manual Operation Annunciator – When lit, the green LED indicates the Freeze Dry unit is being controlled manually by the operator. Each function must be started by the operator.
10. Manual Refrigeration Switch – Used to start only the refrigeration module.
11. Vacuum Annunciator – This green LED indicates that the vacuum pump receptacle on the junction box has power applied.
12. Vacuum Switch – Used to start or stop the vacuum pump.
13. Alarm Annunciator – This red LED indicates that a system Alarm has occurred. Depress the Menu Switch to display the alarm message on the LCD display.

Operation Checklist

The following checklist should be followed prior to each use of your Freeze Dryer:

1. Wipe the interior of the collector chamber with a soft cloth or paper towel to remove any accumulated moisture.
2. Check the collector chamber drain hose to insure 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 collector chamber lid gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the lid gasket to obtain a proper vacuum seal.
4. Remove the accessory drying chamber or manifold from the connection port and using a soft, lint-free cloth or paper towel, wipe the port gasket and sealing surfaces of the drying chamber/manifold to remove any dirt and contaminants that could cause a vacuum leak. Reinstall the drying chamber or manifold on the port. Vacuum grease is not required on the port gasket to obtain a proper vacuum seal.
5. Inspect each sample valve on the drying chamber or manifold and check for any visible damage and for improper installation that might cause a vacuum leak. Also check that each sample valve is closed or in the "vent" position.

Operating the Freeze Dryer

Automatic Start-Up

To run the Auto Mode press the panel switch labeled REFRIGERATION AUTO. The green annunciator above the switch will illuminate. This will start the refrigeration compressor. When the collector reaches –40°C the vacuum pump will start. The Temperature and Vacuum Graph will indicate collector temperature and system vacuum. The LCD display will show the actual temperature of the collector. When the vacuum in the system is above 2000 x 10^{-3} mBar the vacuum display will

indicate "HI." At 2000 x 10^{-3} mBar and below, the display will show the actual vacuum.

When the system vacuum is between 450 and 133 x 10^{-3} mBar, the lower green vacuum graph LED will flash. When the system vacuum level is <133 x 10^{-3} mBar, the green LED will be lit steadily indicating that samples may be added.

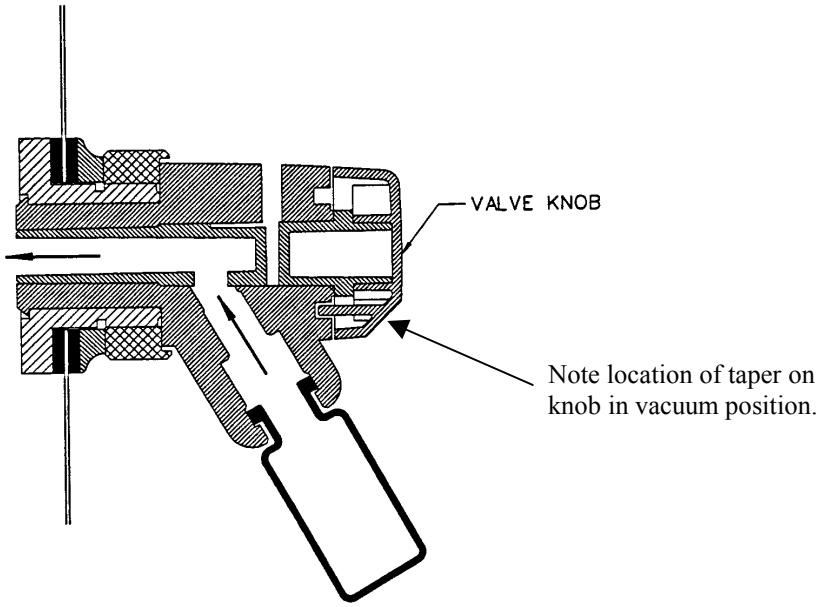
Manual Start-Up

To manually run the freeze dry process press the REFRIGERATION MAN switch. This will start the refrigeration compressor. The green LED above the switch will illuminate. When the collector temperature reaches the desired temperature the vacuum pump may be started by pressing the vacuum switch.

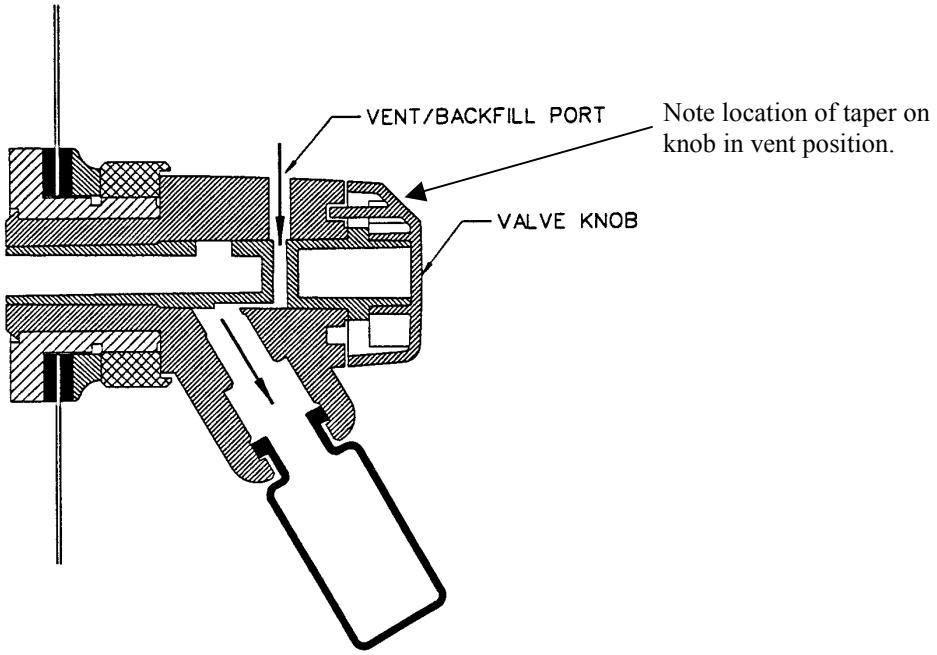
Adding Sample

The following procedure should be followed when freeze drying samples with a drying chamber or manifold:

1. Pre-freeze samples; shell freezing of samples is recommended. Appropriate containers for freeze drying includes ampules, serum bottles, and wide mouth freeze drying flasks. Proper sample container size should always be at least two to three times the sample size (i.e., 150 ml samples should be prepared in 300 ml containers or larger).
2. Connect a pre-frozen sample to a valve on the drying chamber or manifold using an adapter. After connecting a pre-frozen sample to a valve, turn the plastic valve knob to the "VACUUM" position to open the valve which connects the attached sample to system vacuum. The bevel on the knob should be positioned toward the sample port.



3. Before adding another sample, allow system vacuum to return to 133 x 10-3 mBar or lower. 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, the sample is nearly dry. To be certain of low final moisture content, dry the sample for several hours past this point.
5. To remove a container after 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, simply connect the gas supply line to the vent port on the valve. The sample container may now be removed. In the vent position the bevel on the 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.

Alarms and Informational Messages

If any of the following events occur during a run, the alarm LED will illuminate to notify the user.

1. **Power Interruption –** If the power to control system is interrupted either by a power failure or by turning the main power switch off during a freeze dry process, the alarm will be set. When power is restored the freeze dryer will continue to run and the alarm indicator will flash. The display will continue to show the operating parameters of the system. Press MENU to display the alarm "POWER FAIL." Pressing MENU multiple times will identify

other alarms if they have occurred. The alarm indicator can be cancelled by turning the REFRIGERATION off.

2. **Power Line Voltage Out of Range –** If the voltage to the freeze dryer drops or raises beyond a safe level, the alarm indicator will FLASH. Press MENU to display the alarm "LINE VOLTAGE ERROR." Pressing MENU multiple times will identify other alarms if they have occurred. This alarm self cancels when proper voltage is restored.
3. **Temperature Out of Range –** If the temperature of the collector rises above –40°C, the alarm indicator will flash. Press MENU to display the alarm "COLLECTOR TEMPERATURE." Pressing MENU multiple times will identify other alarms if they have occurred. The alarm indicator can be cancelled by turning the REFRIGERATION off.
4. **Service Vacuum Pump –** The vacuum pump normally plugs into the vacuum pump receptacle on the back of the freeze dryer. When the freeze dryer has accumulated a total of 1000 operating hours, the alarm indicator will flash. Press MENU to display the alarm "CHANGE VACUUM PUMP OIL." Pressing MENU multiple times will identify other alarms if they have occurred. This alarm can be cancelled by pressing MENU until the display says "CHANGE VACUUM PUMP OIL" and then pressing VACUUM button and holding until the message disappears. This will reset the 1000 hour timer. It may be necessary to service the vacuum pump more frequently than every 1000 hours depending on the operation of the freeze dryer. The pump oil should be regularly monitored to verify that it is clean.

Shut Down

When a sufficient amount of condensate accumulates on the collector chamber walls, the

collected frozen solvent will obstruct the flow of vapor to the collector chamber. At this point the unit should be defrosted. First, release system vacuum by turning the plastic knob on a valve to the "vacuum"/open position or by pulling the collector chamber drain hose plug. Now turn the vacuum and refrigeration switches on the control panel to "off." When operating in the automatic start-up mode, the vacuum automatically shuts off when the refrigeration switch is turned to "off."

Defrosting

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

1. Pull the collector chamber drain hose out from the side on the unit and remove the drain plug. Place the drain hose in a suitable container to collect the condensate that will be defrosted.
2. Remove the freeze dry chamber or manifold and pour warm water over the ice in the collector chamber. Do not fill the chamber more than 3 inches from the top as water will enter the vacuum stand pipe and drain into the vacuum pump. Dispose of the liquid appropriately.
3. After all condensate is defrosted from the collector chamber, flush the chamber with water and wipe chamber dry.
4. Reinstall the drain hose plug and slide drain hose back into cabinet.

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

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

CHAPTER 5
MAINTAINING YOUR FREEZE DRYER

Under normal operation, the Freeze Dryer requires little maintenance. The following maintenance schedule is recommended:

As needed:

1. Clean up all spills; remove liquids from the chamber.
2. Clean lid and gasket using soft cloth, sponge or chamois and a mild, non-abrasive soap or detergent.
3. Check oil level of the vacuum pump, if applicable. 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.
4. If oil shows cloudiness, particles or discoloration, drain the pump and replace with fresh oil.
5. Utilization of acids requires immediate cleaning and neutralization after a run or physical damage to the collector chamber will result.
6. Check the collection chamber for condensed or frozen solvents and dispose of appropriately. Completely empty the collector chamber before the next run.

Monthly:

1. The rubber components on the Freeze Dryer 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 chamber lid.
3. Using a soft cloth, sponge, or chamois and a mild, non-abrasive soap or detergent, clean the exterior surfaces of the unit. 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 Dryer is operated in a dusty environment, the refrigeration system collector should be cleaned. Using a vacuum cleaner with brush attachment, clean the condenser to ensure proper airflow for peak performance.

CHAPTER 6
MODIFYING YOUR FREEZE DRYER

The operation of your Freeze Dryer can be monitored using either a recorder or a computer. Read this chapter to learn how to:

- connect a recorder to the Freeze Dryer.
- connect a computer to the Freeze Dryer.
- interpret the data received.

Recorder Jack

The system vacuum and collector temperature can be recorded during operation by connecting an appropriate device to the recorder jack on the control panel. The recorder jack is a DIN 8 pin connector. The pin configuration is as follows:

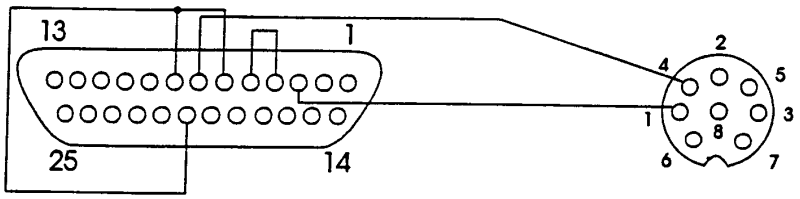
Pin 1 – RS232 Transmit Data
Pin 2 – Blank
Pin 3 – Signal Ground for RS232
Pin 4 – Analog Ground for Recorder
Pin 5 – System Vacuum
Output: 10×10^{-3} mBar = 0 VDC
1024×10^{-3} mBar = 1 VDC

Pin 6 – Collector Temperature
Output: -68°C = 0 VDC
+32°C = 1 VDC
Pin 7 – Blank
Pin 8 – Blank

Computer Connection

Connection diagrams for RS232 interface from Labconco equipment to a computer that supports either a 25 pin D-sub male connector or 9 pin D-sub male connector for serial communication. See *Appendix D: Freeze Dryer Accessories* for available connection cables.

Computer with a 25 pin male serial connector



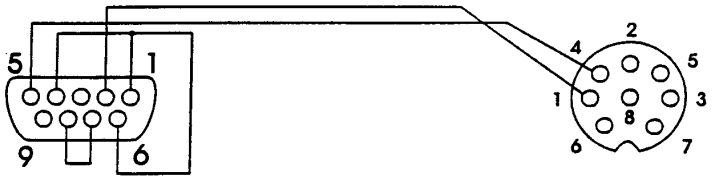
View from back of computer

3-RX (Receive Data)
4-RTS (Request to Send)
5-CTS (Clear to Send)
6-DSR (Data Set Ready)
7-GND (Signal Ground)
8-DCD (Data Carrier Detect)
20-DTR (Data Terminal Ready)

View from front of Labconco Freeze Dryer

1-TXD (Transmit Data)
4-AGND (Analog Ground)

Computer with a 9 pin male serial connector



View from back of computer

1-DCD (Data Carrier Detect)
2-RX (Receive Data)
4-DTR (Data Terminal Ready)
5-GND (Signal Ground)
6-DSR (Data Set Ready)
7-RTS (Request to Send)
8-CTS (Clear to Send)

View from front of Labconco Freeze Dryer

1-TXD (Transmit Data)
4-AGND (Analog Ground)

The purpose of the RS232 interface is to send data to a data collection computer to monitor the state of and activity of the freeze dryer.

This data is half duplex data and is only transmitted from the Freeze Dryer. The data rate and format are listed below:

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 data of the transmitted message is as follows:

1. The following data will be sent with each field separated by a space character.

Freeze Dry Base Status

BASE:<COLLCTR=-86 VAC=0018 RFG=ON VAC=ON

Description

COLLCTR=-86 – indicates the temperature of the collector in degrees Celsius.
VAC=0018 – indicates the vacuum level of the collection system is mBAR x 10^{-3}.
RFG=ON – indicates the state of the refrigeration compressor.
VAC=ON – indicates the state of the vacuum pump.

There are several commercially available software packages which can read this 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 catalog regarding the latest software available.

CHAPTER 7
TROUBLESHOOTING

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

PROBLEM	CAUSE	CORRECTIVE ACTION
Unit will not operate No vacuum	Unit not connected to electrical power	Connect unit to proper electrical power
	Pump not on	Turn on pump
	Pump not connected to unit	Connect pump to unit
	Drain hose plug not installed	Install drain hose plug
	Sample valve open	Close sample valve
	Break or opening in vacuum lines or connections	Locate and repair
Poor vacuum (greater than 500 x 10^{-3} mBar)	Vacuum pump oil level low	Add vacuum pump oil
	Excessive moisture in vacuum pump oil	Change vacuum pump oil
	Vacuum pump gas ballast valve open	Close vacuum pump gas ballast valve

PROBLEM	CAUSE	CORRECTIVE ACTION
Poor vacuum (greater than 500 x 10^{-3} mBar) (cont.)	Leaks in vacuum lines or connections	Locate and repair
	Foreign material on lid gasket	Clean gasket and lid
	Damaged sample valve	Locate and replace

Vacuum Leak Detection

The Freeze Dryer should achieve a vacuum of 133 x 10^{-3} mBar within 10 minutes and should achieve an ultimate vacuum of 33 x 10^{-3} mBar within 18 hours. This extended time interval may be necessary to allow all the components in the system to outgas. After components have outgassed, the pull down time will decrease. To achieve sufficient vacuum, all joints and connections must be tight, the vacuum pump must be operating properly, and the collector temperature must be –40°C or lower. If your Freeze Dryer does not obtain a satisfactory vacuum, the following procedure should be used to locate and correct any vacuum problems.

1. Check each sample valve on the drying chamber or manifold and look for visible damage and for proper installation. To isolate a suspect valve, remove the valve and insert a rubber plug in its place. If the valve proves to be leaking, the plug can be left in place so the drying chamber/manifold can be used until a replacement valve can be obtained.
2. Check vacuum pump oil sight glass. Replace the oil if it is dirty or cloudy; add oil to the pump if the level is low. Close the pump gas ballast valve. Refer to the vacuum pump manufacturer's instructions for further information.
3. Check the collector chamber lid gasket for indentations, cracks or tears. Also clean the gasket using a soft, lint free cloth or paper towel.
4. Check all vacuum hoses and lines for cracks.

5. Check all vacuum connections and joints and tighten any loose hose clamps or fittings.

If any repairs are required on your Freeze Dry System, contact your local laboratory supply dealer. If satisfaction is not obtained through the dealer service network, please call Labconco. Repairs should only be undertaken by a competent technician or through an authorized Labconco service agency.

Refrigeration Module Operation

Under a no-load condition, the FreeZone Plus 2.5 Liter Freeze Dryer can achieve a collector temperature of –84°C or lower, depending on ambient temperature and humidity. If the collector temperature does not reach –84°C within 40 minutes, then the refrigeration module is not functioning properly.

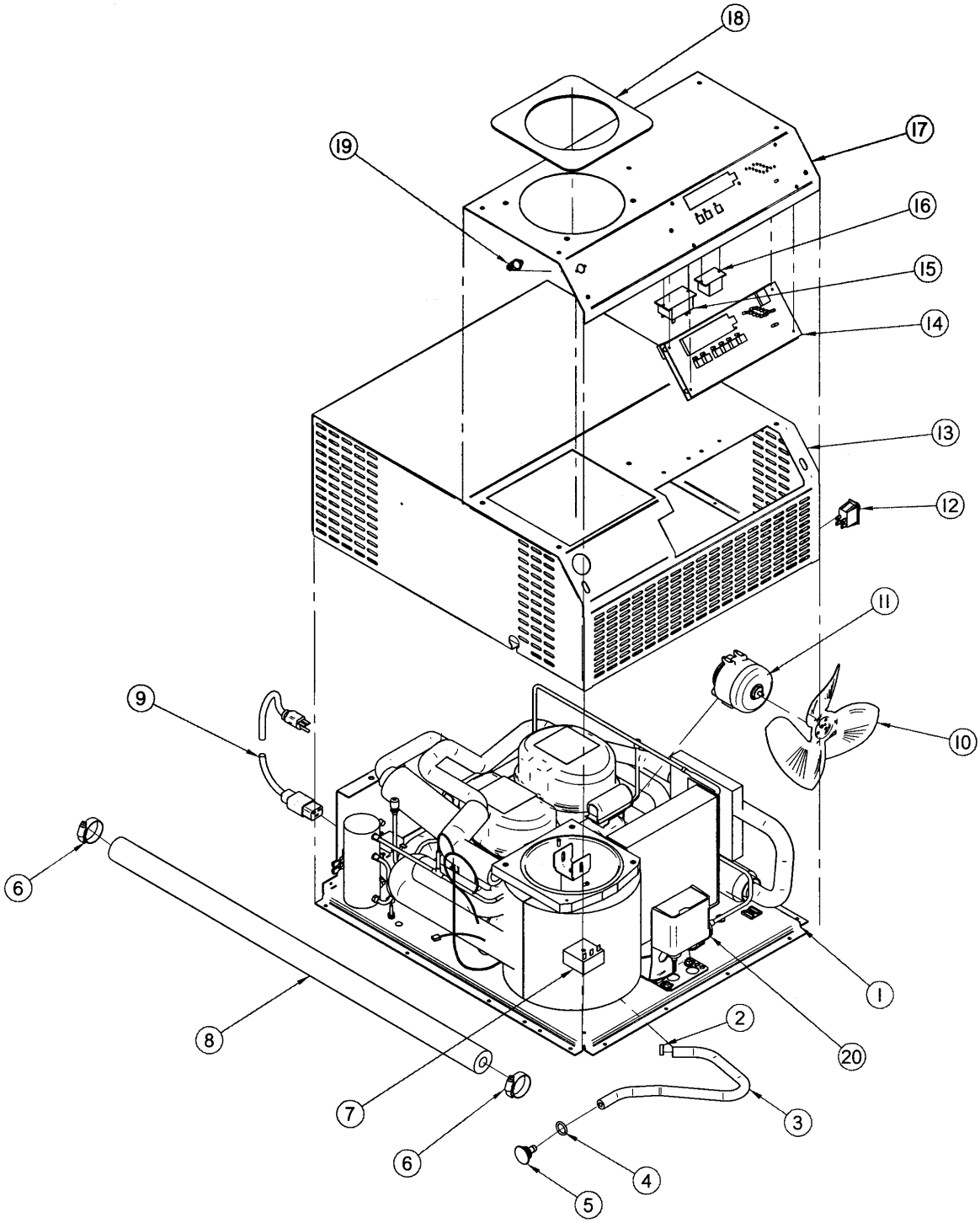
The refrigeration system in your freeze dry unit is a "cascade" type system. Simply, a cascade system is two vapor-compression refrigeration systems in series, connected together via a heat exchanger. Each system has a compressor, condenser, capillary tube metering device and an evaporator. During operation the evaporator of the "high stage" system refrigerates the condenser of the "low stage" system. This takes place in the heat exchanger. The low stage and high stage systems are balanced by design, however, sometimes that balance can be disrupted during operation and the unit will not function properly. If the unit is overloaded, or if the unit is defrosted and restarted in a short time interval, system unbalance is possible. If system unbalance occurs, turn the freeze dry unit off and allow the unit to soak at ambient temperature for 2 to 4 hours.

If any repairs are required on the refrigeration module, contact you local laboratory supply dealer. If satisfaction is not obtained through the dealer service network, please call Labconco. Repairs should only be undertaken by a competent refrigeration technician or through an authorized Labconco service agency.

APPENDIX A
FREEZE DRYER COMPONENTS

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

Item	Quantity	Part No.	Description
1	1	7475600	Condensing Unit (115V)
1A	1	7475601	Condensing Unit (230V)
2	1	1488200	Hose Clamp
3	1	7477300	Drain Tube
4	1	1643600	O-Ring
5	1	772800	Drain Plug
6	2	1967600	Hose Clamp
7	1	7474200	6 Min. Delay Timer (115V)
7A	1	7474400	6 Min. Delay Timer (230V)
8	1	7646000	Vacuum Hose
9	1	1336400	Power Cord (115V)
9A	1	1337300	Power Cord (230V)
10	1	7528900	Fan Blade
11	1	7935100	Fan Motor (115V)
11A	1	7953801	Fan Motor (230V)
12	1	1302300	Rocker Switch
13	1	7470900	Cabinet Weldment
14	1	7515500	PC Board
15	1	1289100	DPST Relay
16	1	1289200	SPST Relay
17	1	7475800	Front Panel Weldment
18	1	7476800	Pot Cover Gasket
19	1	7514500	Recorder Cable Assembly
20	1	7474500	Pressure Switch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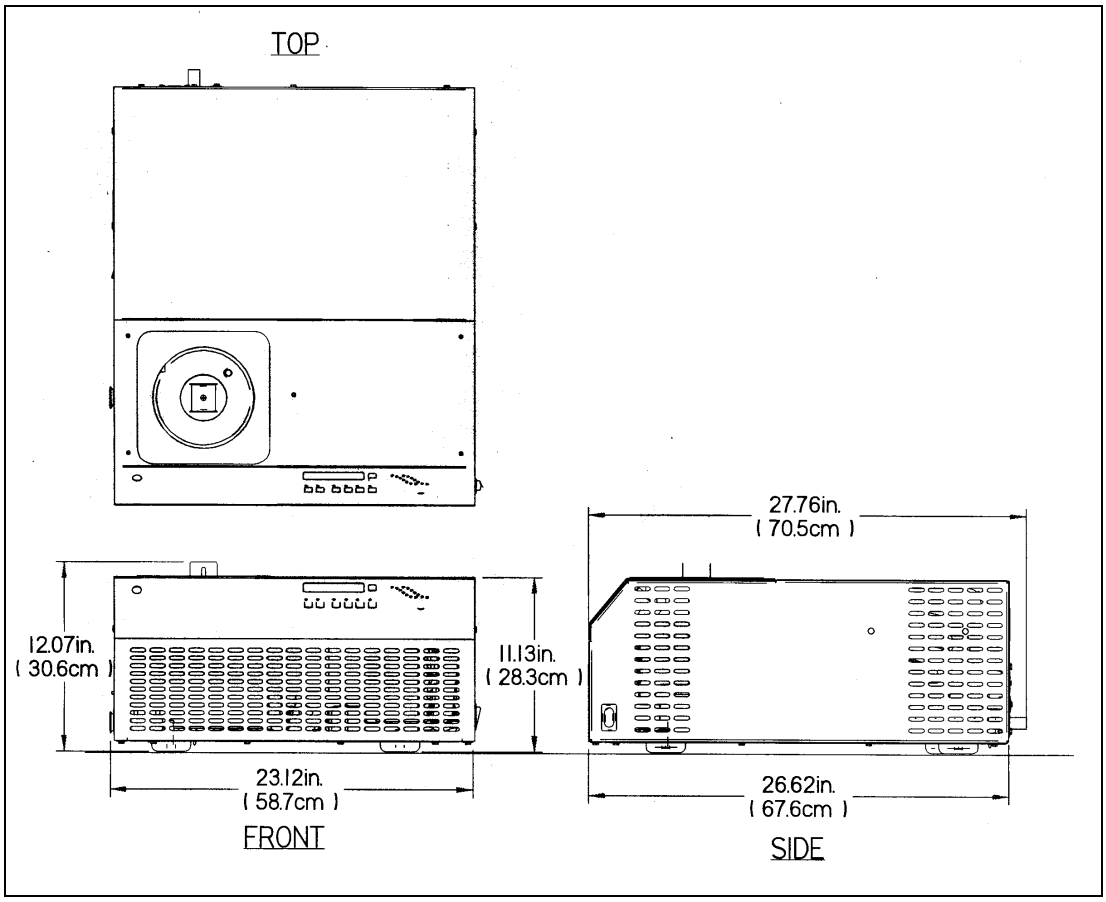
APPENDIX B
FREEZE DRYER
DIMENSIONS



APPENDIX C
FREEZE DRYER SPECIFICATIONS

This Appendix contains technical information about the Freeze Dryer including specifications, environmental operating conditions and wiring diagrams.

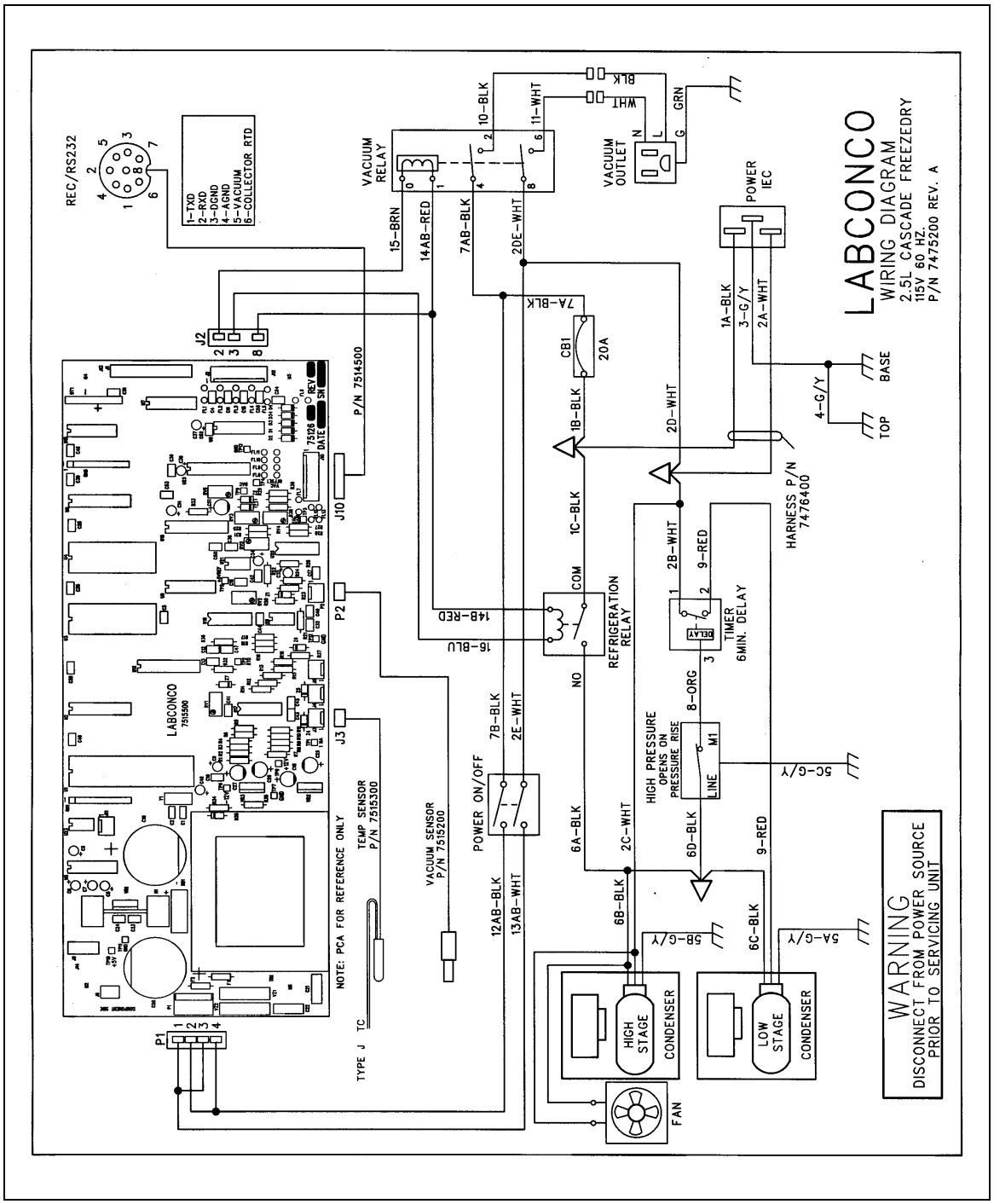
Electrical Specifications

- Nominal amperage for 115V Freeze Dry (model 76700-00) (excluding vacuum pump): 10.5A
- Nominal amperage for 230V Freeze Dry (model 76700-01) (excluding vacuum pump): 5.3A
- Frequency: 115V/60 Hz or 230V/50 Hz
- Phase: Single

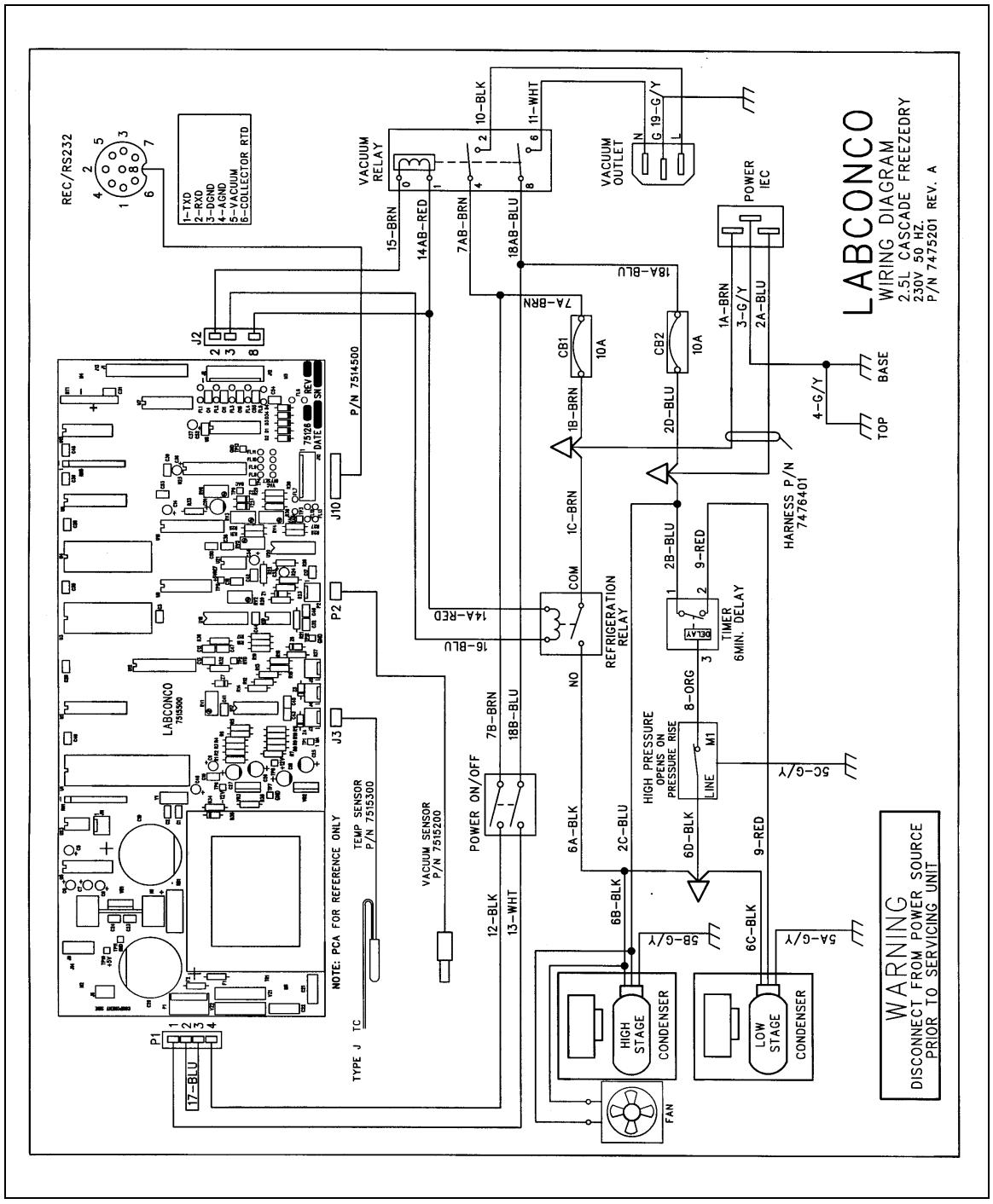
Environmental Conditions

- 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).
- Main supply voltage fluctuations not to exceed ±10% of the nominal voltage.
- Transient overvoltages according to Installation Categories II (Overvoltage 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.

Wiring Diagram (115V Model)



Wiring Diagram (230V Model)



APPENDIX D
FREEZE DRYER ACCESSORIES

The following accessories are available for the Freeze Dryer.

PART #	DESCRIPTION
14677-00	**Vacuum Pump** Two stage direct drive pump, 195 liters/minute. 115 VAC, 50/60 Hz, single phase, 7.8 amps.
77394-03	**Vacuum Pump** Two stage direct drive pump, 195 liters/minute. 230 VAC, 50/60 Hz, single phase, 4.0 amps with reverse IEC plug.
14721-00	**Vacuum Pump** Two stage direct drive pump, 117 liters/minute. 115 VAC, 50/60 Hz, single phase, 4.6 amps.
77394-02	**Vacuum Pump** Two stage direct drive pump, 117 liters/minute. 230 VAC, 50/60 Hz single phase, 2.4 amps with reverse IEC plug.
14722-00	**Pump Inlet Filter** Disposable filter that prevents oil back streaming and protects vacuum pump from submicron particles.

PART #	DESCRIPTION
14723-00	**Pump Exhaust Filter** Disposable filter that removes visible oil mist from vacuum pump exhaust.
77720-00	**Soda Acid Trap** Secondary trap that prevents migration of corrosive chemicals into vacuum pump.
77721-00	**Replacement Media for Soda Acid Trap**
77725-00	**Charcoal Solvent Trap** Secondary trap that prevents migration of organic solvents into vacuum pump.
77726-00	**Replacement Activated Charcoal Media for Charcoal Solvent Trap**
75380-00	**Secondary Vacuum Trap** 9 ¾" high x 7 7/8" diameter, 304 stainless steel with ¾" vacuum connections.
75096-00	**Sample Valve Kit** Includes Neoprene valve body, knob and installation parts.
75228-00	**12 Port Chamber** 9 ¾" high x 7 7/8" diameter, 304 stainless steel with 12 freeze dry valves.
78670-00	**Clear Drying Chamber** 13" high x 13" diameter clear acrylic chamber.

PART #	DESCRIPTION
75210-00	**Heated Drying Chamber** 10 ½" high x 9" diameter type 304 stainless steel. The 3 shelf product heaters heat to 43°C (110°F). 115V
75210-01	**Heated Drying Chamber** 10 ½" high x 9" diameter type 304 stainless steel. The 3 shelf product heaters heat to 43°C (110°F). 230V
75092-00	**Product Heater** For use in chamber 75228-00. Provides 3 heated shelves operating at 43°C (110°F). 115V
75092-01	**Product Heater** For use in chamber 75228-00. Provides 3 heated shelves operating at 43°C (110°F). 230V
75223-00	**20 Port Manifold** 10" high x 27 ½" wide x 8 5/8" deep. Type 304 stainless steel manifold with 20 freeze dry valves. Requires attachment port lid accessory 74774-00.
78685-00	**48 Port Ampule Manifold** 28 5/8" high x 5" wide x 5" deep. Type 304 stainless steel with 48 each ¼" tube stems.
75340-00	**Cable** Connects the RS 232 output from the Freeze Dryer (or Freeze Dryer/Tray Dryer combination) to an IBM compatible computer with a 9-pin serial data port.
75341-00	**Cable** Connects the RS 232 output from the Freeze Dryer (or Freeze Dryer/Tray Dryer combination) to an IBM compatible computer with a 25-pin serial data port.

Fast-Freeze® Flasks

Lyph-Lock® Flasks

Serum Bottles & Vials

Ampules

DECLARATION OF CONFORMITY

Application Council Directive(s): 73/23/EEC, 89/336/EEC

Standard(s) to which conformity is declared: EN61010, EN55022, EN50082-1

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 – Freeze Dryer
Model No.:	775 followed by two digit number based on model size; followed by another two digit number based on options. 77400 followed by two digit number based on options. 79340 followed by two digit number based on options. 76700 followed by two digit number based on options. 79600 followed by two digit number based on options.
Serial No.:	Various – See Individual Declaration

Year of Manufacture: 1995 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)

*An individual version of this declaration is included with your shipping/customs documentation.

Labconco P/N 36960-02, Rev. C, ECO B296