

FreeZone[®] 4.5 Liter Benchtop
Freeze Dry System
Model 77500

INSTRUCTION MANUAL

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Components Shipped

Carefully check the contents of the carton for damage that might have occurred in transit. Do not discard the carton or packaging material until all components have been checked against the following component list and the equipment has been installed and tested:

As shipped, the carton should contain the following:

Part Number	Description
77500-xx	4.5 Liter Benchtop Freeze Dry System
76885	Lid Gasket
77307	Condenser Chamber Lid
76460	Vacuum Hose
19676	Hose Clamp
75198	Instruction Manual
13373 or 13364	Power Cord (Model 77500-01 and 77500-04) Power Cord (Model 77500-00 and 77500-03)
12910	Plug (Model 77500-01 and 77500-04)

INTRODUCTION

General Description

The Labconco FreeZone® 4.5 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. Shown below is a 4.5 liter benchtop unit.

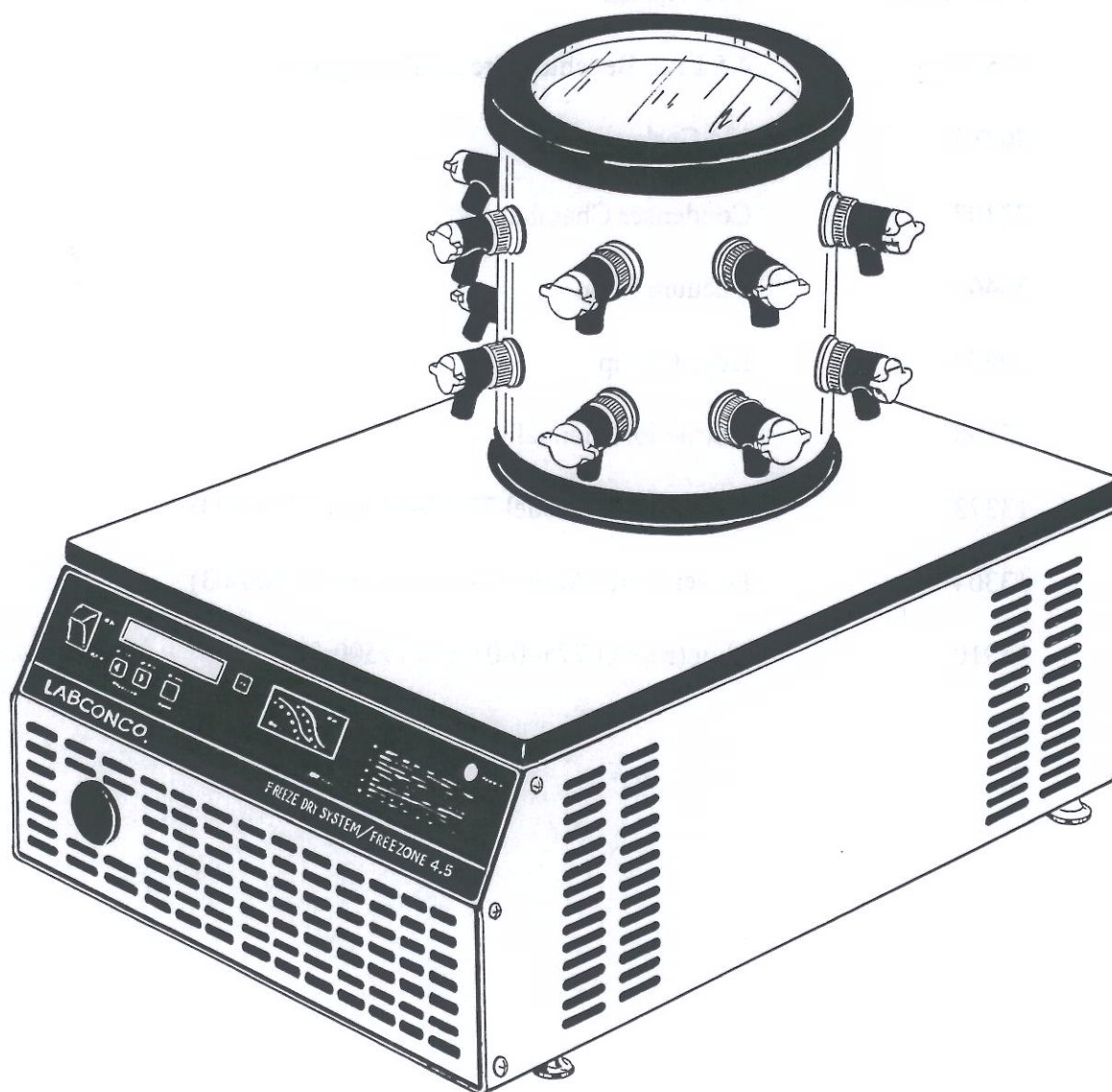


Figure 1

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 condenser; the removal of heat, due to ice formation, from the condenser 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 condenser 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 condenser 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.

SAFE TEMPERATURE AND DRYING TIMES FOR SELECTED MATERIALS			
Material 10MM Thick	Safe Temperature °C	Condenser 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.

Table 1

INTRODUCTION

Freeze Dry Rates (Con't)

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 condenser and the vacuum pump. The condenser, which should be at least 10 to 15°C colder than the eutectic temperature (melting temperature) of the sample, traps water vapor as ice. Since the vapor pressure at the condenser is less than that of the sample, the flow of water vapor is from the sample to the condenser. 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

Freeze Dry Capacity (Con't)

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 condenser 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 $133 \times 10^{-3}\text{mBar}$ 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 condenser temperature can be cooled to below -75°C , or a dry ice/solvent trap can be used between the condenser 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.

INTRODUCTION

Component Identification (See Figure 2)

- (1) **Cabinet.** The cabinet is epoxy coated steel for strength and durability.
- (2) **Control Panel.** The control panel contains readouts for vacuum and temperature and contains all control switches. The control panel also contains a recorder jack.
- (3) **Cabinet Top.** The cabinet top is corrosion resistant and smooth.
- (4) **Condenser Chamber Lid.** The lid is clear acrylic so that the amount of condensate may be monitored.
- (5) **Condenser Chamber Lid Gasket.** Molded of neoprene, the gasket forms a tight seal between condenser chamber and lid.
- (6) **Condenser Chamber.** Constructed of 304 stainless steel, it contains the stainless steel collector coil and vacuum stand pipe.
- (7) **Condenser Chamber Drain Hose.** The drain hose extends from the control panel for draining condensate.
- (8) **Drain Plug.**
- (9) **Refrigeration Module.** The system is a mechanical, capillary tube design.
- (10) **Vacuum Hose with clamps.** The hose connects the vacuum pump to the freeze dry unit.
- (11) **Sample Valve.** Pliable neoprene valves accommodate virtually every type of freeze dry glassware.
- (12) **Valve Stem.** Polypropylene stem directs vacuum to the sample. It permits the sample to be vented or to be backfilled with dry sterile air or dry nitrogen.
- (13) **Back Panel.** The panel is removable to provide access to the inside of the cabinet.

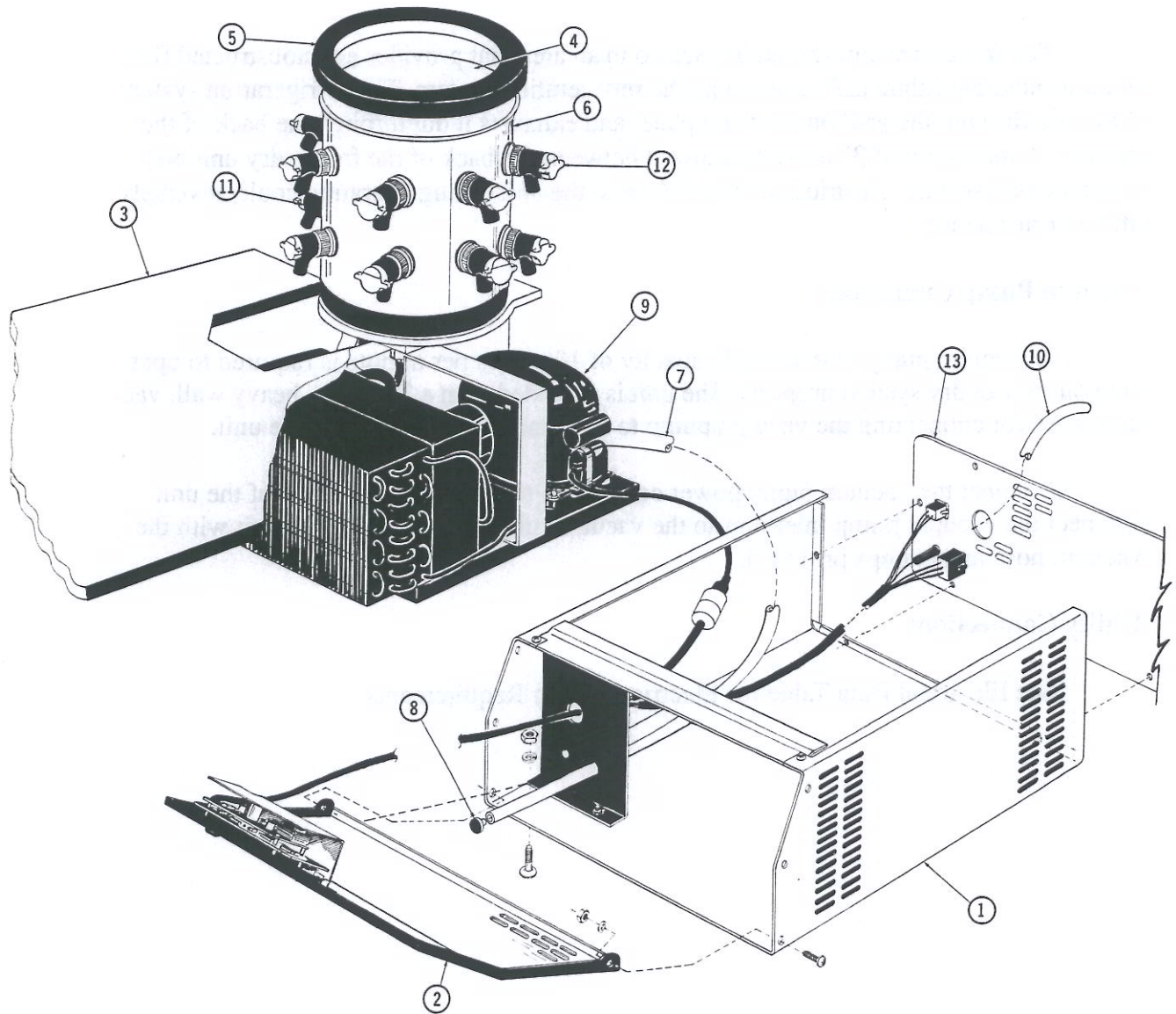


Figure 2

INSTALLATION

Preparation

Remove the back panel from the freeze dry unit and inspect all refrigeration components for visible damage. Check to see that the condenser fan on the refrigeration module spins without obstruction.

Location

The freeze dry unit 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 of the cabinet. A minimum of 3" must be allowed between the back of the freeze dry unit and adjacent wall surface. Restriction of airflow into the unit during operation could adversely affect performance.

Vacuum Pump Connection

A vacuum pump with a rated capacity of 100 liters per minute is required to operate your freeze dry system properly. The unit is provided with a 3/4" I.D., heavy wall, vacuum hose for connecting the vacuum pump to the condenser chamber on the unit.

Connect the vacuum pump power cord to the receptacle on the back of the unit. Connect the vacuum pump inlet port to the vacuum tube on the back of the unit with the vacuum hose and clamps provided.

Utility Connections

See Electrical Data Table for Electrical Power Requirements.

SAFETY PRECAUTIONS

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

Do not attempt to chip ice off of the collector coil as serious damage to the refrigeration module may result. The release of refrigerant could injure your eyes.

NORMAL OPERATION

Notes

Operation Checklist

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

- (1) Wipe the interior of the condenser chamber with a soft cloth or paper towel to remove any accumulated moisture.
- (2) Check the condenser 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 condenser 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) Inspect each sample valve on the drying chamber 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.

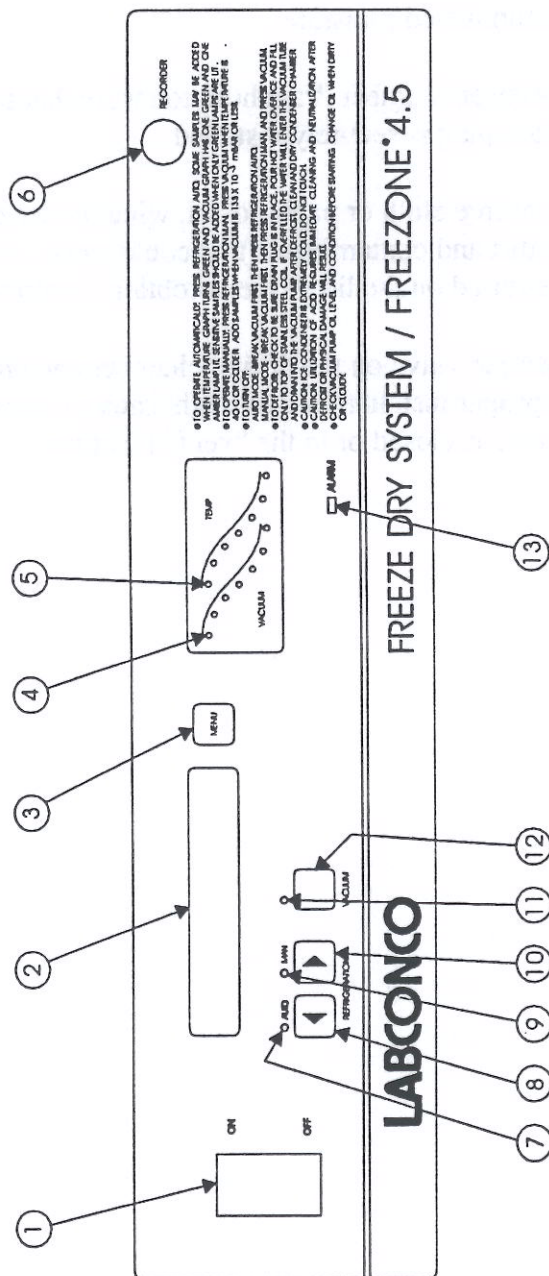


Fig. 3

Control Panel Identification (See Figure 3)

- (1) **Main Power Switch.** Turns the unit on or off.
- (2) **LCD Display.** Displays system parameters and alarm messages. Parameters displayed are system vacuum (10^{-3} mBar), and collector temperature ($^{\circ}\text{C}$). 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 2000×10^{-3} mBar. The indicators will sequence down when the vacuum level reaches 2000, 1000, 800, 600, 450, 133×10^{-3} mBar. The lower green LED will flash when the system vacuum level is 450 to 133×10^{-3} mBar and illuminate steady below 133×10^{-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; 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 has power applied.

NORMAL OPERATION

- (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.

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

When the system vacuum is between 450 and 133×10^{-3} mBar, the lower green graph LED will flash. When the system vacuum level is $<133 \times 10^{-3}$ mBar, the green LED will be lit steady 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.

Alarms & Informational Messages

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

1. Power Interruption

If the power to the 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 had 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 had occurred. This alarm self cancels when proper voltage is restored.

NORMAL OPERATION

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 TEMP ERROR'. Pressing MENU multiple times will identify other alarms if they had 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 in 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 had occurred. This alarm can be cancelled by pressing MENU until the display says "NO ALARMS" and then pressing VACUUM button and holding until the alarm indicators stops flashing. 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.

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 - RS 232 Transmit Data

Pin 2 - Blank

Pin 3 - Signal Ground for RS 232

Pin 4 - Analog Ground for Recorder

Pin 5 - System Vacuum

Output: $10 \times 10^{-3} \text{ mBar} = 0\text{VDC}$

$1024 \times 10^{-3} \text{ mBar} = 1\text{VDC}$

Pin 6 - Collector Temperature

Output: $-68^{\circ}\text{C} = 0 \text{ VDC}$

$+32^{\circ}\text{C} = 1 \text{ VDC}$

Pin 7 - Blank

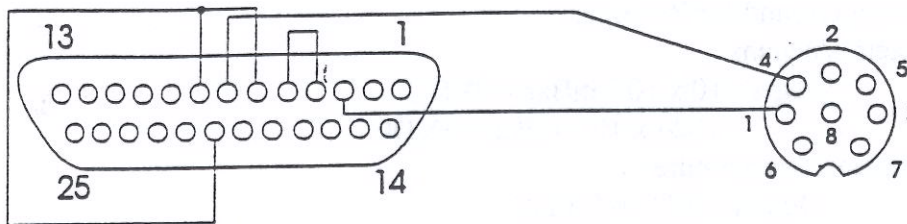
Pin 8 - Blank

NORMAL OPERATION

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.

Check your computer to see which type of serial port is provided, then wire a connecting cable according to the appropriate diagram below:

1. Computer with a 25 pin male serial connector.



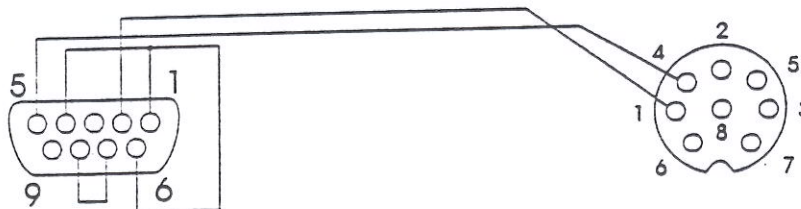
View from back of computer

View from front of Labconco Freeze Dry equipment

- 2 - TX (Transmit Data)
- 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)

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

2. Computer with a 9 pin male serial connector



View from back of computer

View from front of Labconco Freeze Dry equipment

- 1 - DCD (Data Carrier Detect)
- 2 - RX (Receive Data)
- 3 - TX (Transmit 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)

- 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 dry unit. 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 content of the transmitted message is as follows:

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

Freeze Dry Base Status

BASE:<COLLCTR=-41 VAC=0018 SHELL=-43 RFG=ON VAC=ON PRG=OFF SHL=ON DFRST=OFF>

Description:

COLLCTR=-41 - indicates the temperature of the collector in degrees Celsius.

VAC=0018 - indicates the vacuum level of the collection system is mBAR x 10⁻³.

SHELL=-43 - indicates the temperature of the shell freezer if the option is present.

RFG=ON - indicates the state of the refrigeration compressor.

VAC=ON - indicates the state of the vacuum pump.

PRG=OFF - indicates the state of the purge valves if the option is present.

SHL=ON - indicates the state of the Shell Freezer compressor if the option is present.

DFRST=OFF - indicates the state of the defrost heater if the option is present.

If a Stoppering Tray Dryer is used with the Base unit, the status from the Tray Dryer will be issued with Base units status also. See Tray Dryer manual for details on status contents.

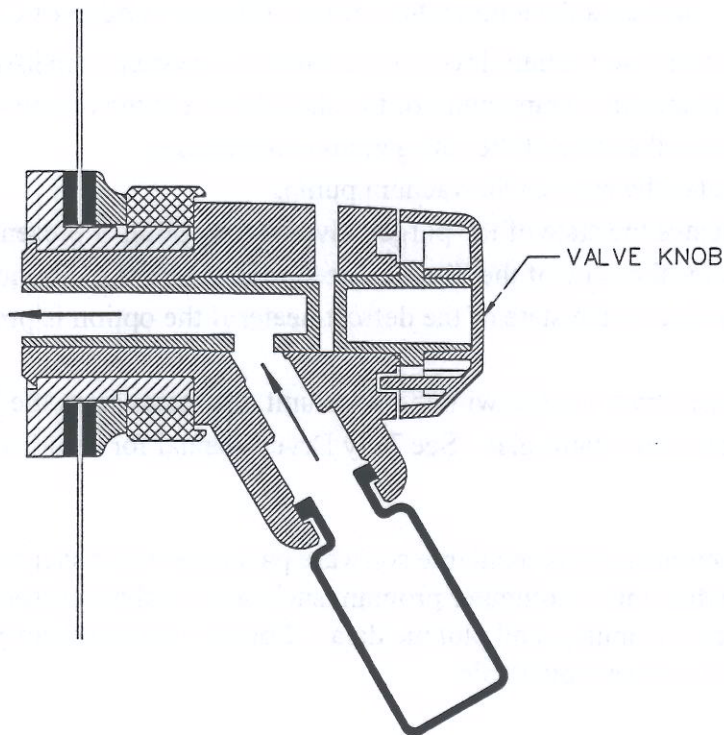
There are several commercially available software packages which can read this RS 232 data and enter the data into a computer program such as a word processor (to create a text file) or spreadsheet (to tabulate and plot the data). Consult your laboratory supply catalog regarding the latest software available.

NORMAL OPERATION

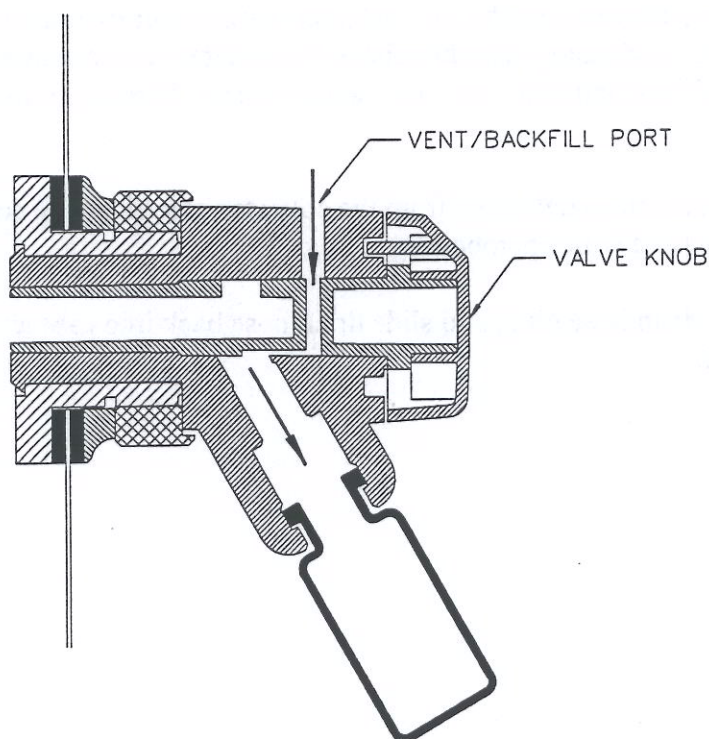
Adding Sample

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 include ampoules, 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. 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 133×10^{-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.

NORMAL OPERATIONS

Shut Down

When a sufficient amount of condensate accumulates on the collector coil, the collector temperature will not remain below -40°C . 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 condenser 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 coil:

- (1) Pull the condenser chamber drain hose out from the control panel on the unit and remove the drain plug. Place the drain hose in a suitable container to collect the condensate that will be defrosted off the collector coil.
- (2) Remove the condenser chamber lid and pour warm water over the collector coil. Do not fill the condensing chamber above the collector coil 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 coil, flush the condenser chamber with water and wipe chamber dry.
- (4) Reinstall the drain hose plug, and slide drain hose back into cabinet through the control panel.

ROUTINE MAINTENANCE SCHEDULE

Under normal operation, your freeze dry unit will require little routine maintenance. The following maintenance schedule is recommended:

Weekly

- (1) Check the condition and level of the vacuum pump oil. If the oil level is low, then add oil. If the oil contains excessive amounts of moisture, detected by a cloudiness in the oil, it will be necessary to change the oil. For further information regarding procedures for changing the vacuum pump oil, refer to the vacuum pump manufacturer's instructions. To expel small amounts of water that may have accumulated in the pump oil, periodically operate the vacuum against a dry, tight system. The gas ballast must be open.

Monthly

- (1) The rubber components on the freeze dry unit may eventually deteriorate and require replacement. The effective life of rubber parts will depend 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 condenser chamber lid.
- (3) Using a soft cloth, sponge, or chamois and a mild, non-abrasive soap or detergent, clean the top, side, and front panels of the unit. Liquid spray cleaners and polishes may be used on the side and front panels. Do not use solvents to remove stains from the panels as they may damage the finish.
- (4) All weekly activities.

Semi-Annually

- (1) Using a vacuum cleaner with brush attachment, clean the refrigeration module condenser to ensure proper air flow for peak performance. More frequent cleaning may be required if the freeze dry unit is operated in a dusty environment.
- (2) All monthly activities.

VACUUM LEAK DETECTION

The freeze dry unit should achieve a vacuum of 133×10^{-3} mBar within 10 minutes and should achieve an ultimate vacuum of 33×10^{-3} mBar within 18 hours. This extended time interval may be necessary to allow all the components in the system to out-gas. 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 dry unit 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 condenser chamber lid gasket for indentions, 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 at (800) 821-5525 or (816) 333-8811. Repairs should only be undertaken by a competent technician or through an authorized Labconco service agency.

Under a no-load condition, your freeze dry unit can achieve a collector temperature of -40°C or lower, depending on ambient temperature and humidity. If the collector temperature does not reach -40°C within 20 minutes, then the refrigeration module is not functioning properly.

If any repairs are required on the refrigeration module, contact your local laboratory supply dealer. If satisfaction is not obtained through the dealer service network, please call Labconco at (800) 821-5525 or (816) 333-8811. Repairs should only be undertaken by a competent refrigeration technician or through an authorized Labconco service agency.

FREEZE DRY UNIT SERVICE

Back Panel Removal

- (1) While supporting the back panel, loosen and remove the screws securing the back panel to the cabinet.

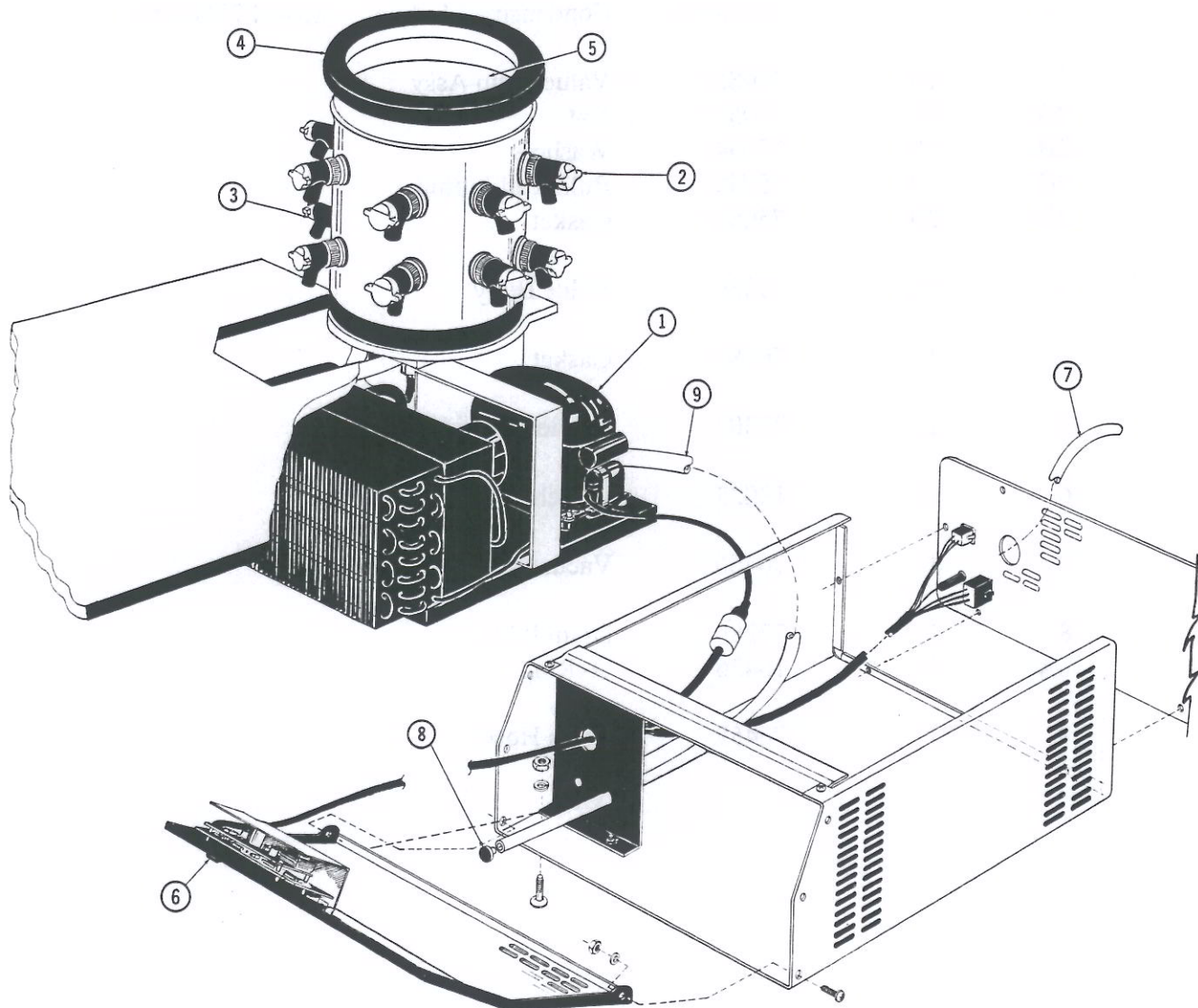
Control Panel Removal

- (1) Unplug the unit or disconnect it from power.
- (2) Loosen and remove the top two screws at each end of the control panel which secure it to the cabinet.
- (3) Loosen the bottom screw at each end of the control panel and tilt the panel down.
- (4) Disconnect the power harness, the vacuum sensor harness, and the temperature sensor harness from the back of the control panel.
- (5) Remove the bottom screw at each end of the control panel and lift control panel away from unit.

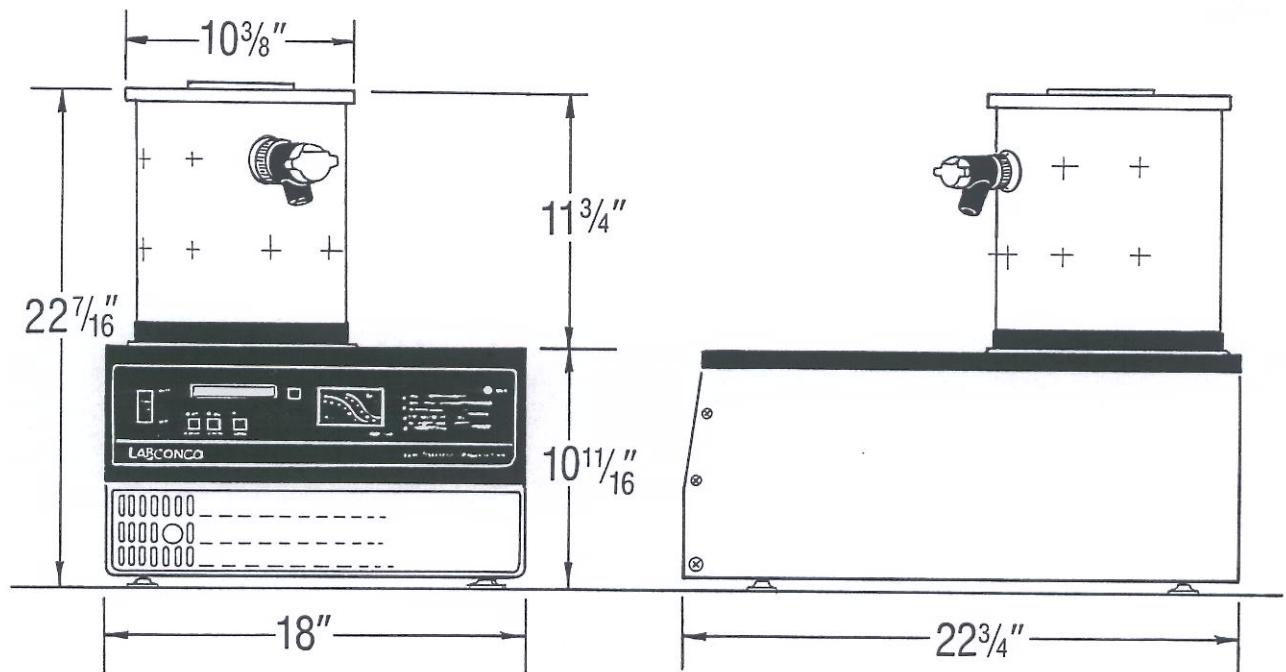
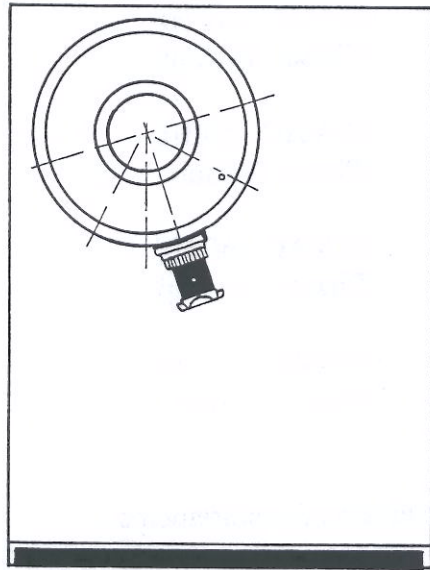
REPLACEMENT PARTS

ITEM	QTY	PART NO.	DESCRIPTION	MODEL
1	1	75253-00	Condensing Module	Model 77500-00
1A	1	75253-01	Condensing Module	Model 77500-01
1B	1	75253-02	Condensing Module	Model 77500-03
1C	1	75253-03	Condensing Module	Model 77500-04
2	10	75086	Value Stem Assy	
2A	10	75053	Nut	
2B	10	75054	Washers	
2C	10	75055	Bulkhead Fitting	
2D	20	75058	Gasket	
3	10	75056	Value Body	
4	1	76885	Gasket	
5	1	77307	Condenser Lid	
6	1	13023	Switch	
7	1	76460	Vacuum Hose	
8	1	77280	Drain Plug	
8A	1	16436	O- Ring	
9	1	76459	Drain Hose	

REPLACEMENT PARTS



FREEZE DRY UNIT DIMENSIONS



ELECTRICAL DATA

MODEL NUMBER	ELECTRICAL REQUIREMENTS
77500	115 VAC - 60 Hz 1 Phase - 14 Amp
77500-01	230 VAC - 50 Hz 1 Phase - 8 Amp
77500-03	115 VAC - 60 Hz 1 Phase - 14 Amp
77500-04	230 VAC - 60 Hz 1 Phase - 8 Amp

All electrical specifications include vacuum pump electrical requirements.

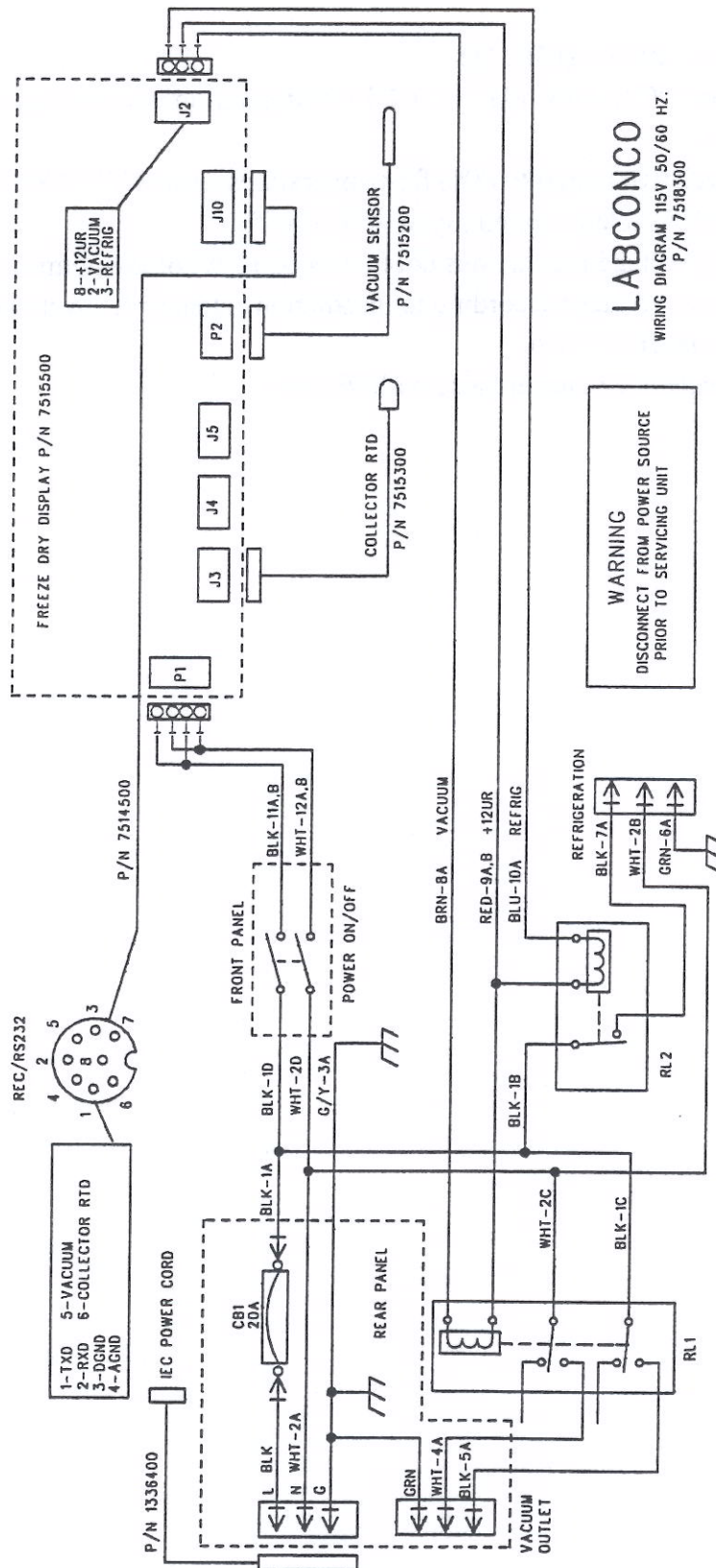
The vacuum pump maximum power requirements should be 115V, 7A or 230V,
4.5A.

The Freeze Dryer is designed to operate safely **under the following conditions:**

- Indoor use
- Altitude up to 2000M (6562 Ft)
- Temperature 5°C to 40°C (41° to 104°F) Refrigeration will meet specs as stated on page 28
- Maximum relative humidity 80% for temperatures up to 31°C (88°F) decreasing linearly to 50% relative humidity at 40°C (104°F)
- Main supply voltage fluctuations not to exceed $\pm 10\%$ of the nominal voltage
- Transient over voltages according to installation category II (over voltage categories per IEC 1010)
- Pollution degrees 2 in accordance with IEC 464

WIRING DIAGRAMS

Models 77500-00 and 77500-03
(115 Vac, 60 Hz, 1 Phase)



TROUBLESHOOTING

PROBLEM	CAUSES	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 10⁻³mBar)	Vacuum pump oil level low	Add vacuum pump oil
	Excessive moisture in vacuum pump oil	Replace vacuum pump oil
	Vacuum pump gas ballast valve open	Close vacuum pump gas ballast valve
	Leaks in vacuum lines or connections	Locate and repair
	Foreign material on lid gasket	Clean gasket and lid
	Damaged sample valve	Locate and replace

If you are having problems with the operation of your freeze dry unit, call Labconco at (800) 821-5525 or (816) 333-8811.

We are committed to providing our customers with quality equipment and service after the sale. Part of this objective involves keeping you informed of changes and new product additions. We therefore request that you take a moment to fill out the product registration card so we may know your location as well as some of the reasons that prompted you to purchase our products.

Labconco Corporation warrants products of its manufacture for one year, from receipt of the equipment by the purchaser, against defects in materials and workmanship. This limited warranty covers parts and labor but not transportation and insurance charges. In the event of a warranty claim contact 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 circumstance shall Labconco Corporation be liable for indirect, consequential or special damages of any kind.** This statement of warranty 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 expendable items such as filters are not covered by this warranty. Damage due to corrosion or accidental breakage are also not covered.

WARNING: 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 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.

SHIPPING CLAIMS

If a shipment is received in visibly damaged condition, be certain to make a notation on the delivering carrier's receipt and have his agent confirm the damage on your receipt. Otherwise, the damage claim may be refused.

If concealed damage or pilferage is discovered, notify the carrier immediately and retain the entire shipment intact for inspection. Interstate Commerce Commission rules require that the claim be filed with the carrier within 15 days after delivery.

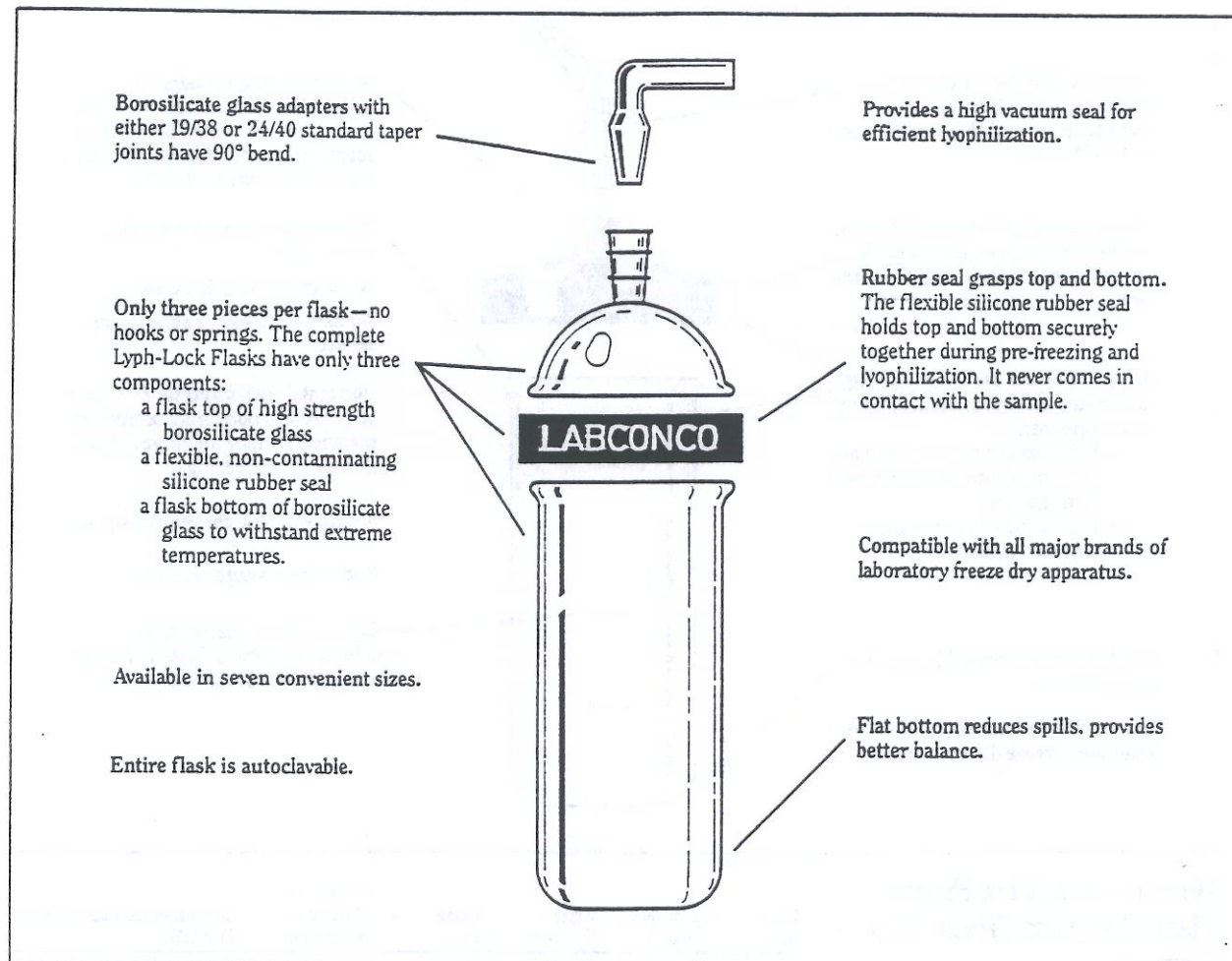
NOTE: Do not return goods. Goods returned without prior authorization will not be accepted. Labconco Corporation and its dealers are not responsible for shipping damage. Claims must be filed directly with the freight carrier by the recipient. If authorization has been received to return this product, by accepting this approval, the user assumes all responsibility and liability for biological and chemical decontamination and cleansing. Labconco reserves the right to refuse delivery of any products which do not appear to have been properly cleaned and/or decontaminated prior to return.

Accessory Part #	Description
14677	Vacuum Pump Two stage direct drive pump, 195 liters/minute. 115 VAC, 50/60 Hz single phase, 7.2 amps.
77394-01	Vacuum Pump Two stage direct drive pump, 195 liters/minute. 230 VAC, 50/60 Hz, single phase, 4.0 amps.
14721	Vacuum Pump Two stage direct drive pump, 117 liters/minute. 115 VAC, 50/60 Hz, single phase, 4.6 amps.
77394	Vacuum Pump Two stage direct drive pump, 117 liters/minute. 230 VAC, 50/60 Hz, single phase, 2.4 amps.
14722	Pump Inlet Filter Disposable filter that prevents oil back streaming and protects vacuum pump from submicron particles.
14723	Pump Exhaust Filter Disposable filter that removes visible oil mist from vacuum pump exhaust.
77720	Soda Acid Trap Secondary trap that prevents migration of corrosive chemicals into vacuum pump.
77721	Replacement Soda Salt Media For Soda Acid Trap
77725	Charcoal Solvent Trap Secondary trap that prevents migration of organic sol- vents into vacuum pump.
77726	Replacement Activated Charcoal Media For Char- coal Solvent Trap
75380	Secondary Vacuum Trap 9-3/4" high x 7-7/8 diameter, 304 stainless steel with 3/4" vacuum connections.

ACCESSORIES

- 75382 **Secondary Vacuum Trap**
9-3/4" high x 7-7/8" diameter, 304 stainless steel with
1/2" vacuum connections.
- 75384 **Secondary Vacuum Trap**
7-7/8" high x 6-5/8" diameter, 304 stainless steel with
1/2" vacuum connections
- 75096 **Sample Valve Kit**
Includes Neoprene valve body, knob and installation
parts.
- 75223 **24 Port Manifold**
10" High x 27-1/2" Wide x 8-5/8" Deep. Type 304
stainless steel manifold with 24 freeze dry valves.
Requires accessory attachment port lid 77628.
- 75340 **Cable** to connect 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 **Cable** to connect 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.

Lyph-Lock® Flasks have easy-to-use features.



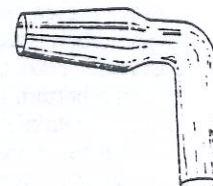
How to select Lyph-Lock Flasks for your Freeze Dry System.

Select the Lyph-Lock Flasks based on your sample sizes. Flasks should be filled to 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.

Flask Size	Complete Flask 19/38 STJ	Complete Flask 24/40 STJ	Flask Top 19/38	Flask Top 24/40	Lyph-Lock Seal	Flask Bottom	Dimensions/Flask Bottom H x I.D.
25 ml	75500	75540	75520	75560	75590	75570	37 mm x 34 mm
50 ml	75502	75542	75520	75560	75590	75572	67 mm x 34 mm
100 ml	75504	75544	75522	75562	75592	75574	50 mm x 59.2 mm
250 ml	75506	75546	75522	75562	75592	75576	110 mm x 59.2 mm
500 ml	75508	75548	75524	75564	75594	75578	103 mm x 90.2 mm
750 ml	75509	75549	75524	75564	75594	75579	145 mm x 90.2 mm
1000 ml	75510	75550	75524	75564	75594	75580	187 mm x 90.2 mm

Accessories

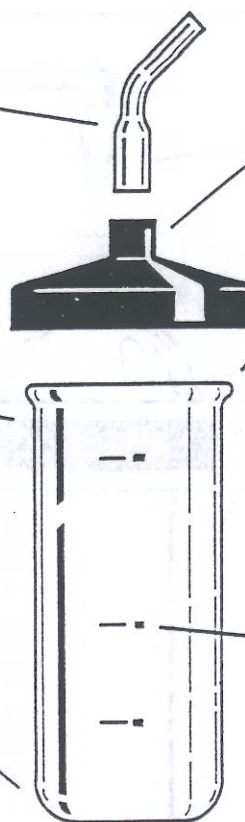
Add the Adapters for connecting the Lyph-Lock Flasks to the valve ports on your drying chamber or manifold.



90° Bend Adapter	Description
75680	Connects 19/38 STJ Flask Top to 1/2" valve
75682	Connects 19/38 STJ Flask Top to 3/4" valve
75684	Connects 24/40 STJ Flask Top to 1/2" valve
75686	Connects 24/40 STJ Flask Top to 3/4" valve

ACCESSORIES

Fast-Freeze® Flasks simplify freeze drying.



Variety of adapters available. You may select from glass or stainless steel adapters available straight and with 45° bend.

Top seals easily. The flexible silicone rubber top snaps on and off easily, yet provides a reliable, high vacuum seal.

Only two pieces per flask. The complete Fast-Freeze Flask has only two components:
 a high strength borosilicate glass bottom to withstand extreme temperatures
 a flexible, non-contaminating silicone rubber top.

Flat bottom reduces spills, provides better balance.

Compatible with all major brands of laboratory freeze dry apparatus.

No complex filter retainer is necessary. For those who use filters, they are easily inserted between the adapter and top of the flask. Filters are supplied with each flask.

Wide mouth opening loads fast, cleans easily.

No vacuum grease is necessary.

No threads, hooks or springs are needed.

Horizontal shell freezing. Flask may be placed in a horizontal position in shell freezer, allowing more surface area for faster freezing.

Available in nine convenient sizes.

Entire flask is autoclavable.

Convenient volumetric guides indicate quantity in flask, and aid in reconstitution of sample.

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

Select the Fast Freeze Flasks based on your sample sizes. Flasks should be filled to 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 and retainer rings. Tops, bottoms and filter paper are available separately as replacement components.

Flask Size	Complete Flask	Flask Bottom	Flask Top	Flask Top/Adapter Diameter	Dimensions/Flask Bottom H x I.D.
40 ml	75400	75420	75440	½"	76 mm x 34 mm
80 ml	75402	75422	75440	½"	115 mm x 34 mm
120 ml	75403	75423	75442	¾"	68 mm x 59.2 mm
150 ml	75404	75424	75442	¾"	85 mm x 59.2 mm
300 ml	75406	75426	75442	¾"	145 mm x 59.2 mm
600 ml	75408	75428	75444	¾"	135 mm x 90.2 mm
900 ml	75409	75429	75444	¾"	190 mm x 90.2 mm
1200 ml	75410	75430	75444	¾"	240 mm x 90.2 mm
2000 ml	75412	75432	75444	¾"	380 mm x 90.2 mm

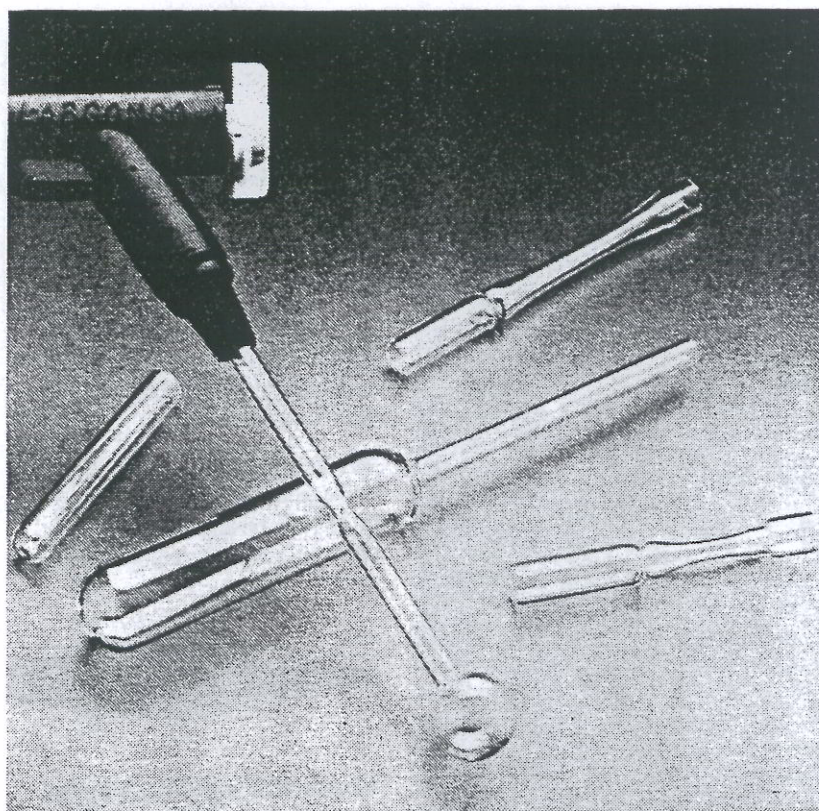
Accessories

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 ½" and ¾" diameters.

75448-10 FILTER PAPER
 Package of 1000.

Diameter	Straight Adapter	45° Bend Adapter
Borosilicate Glass		
½" flask top to ½" valve	75450	75456
¾" flask top to ¾" valve	75452	75458
½" flask top to ¾" valve	75454	75460
¾" flask top to ½" valve	75454	75460
Stainless Steel		
½" flask top to ½" valve	75470	75474
¾" flask top to ¾" valve	75472	75476

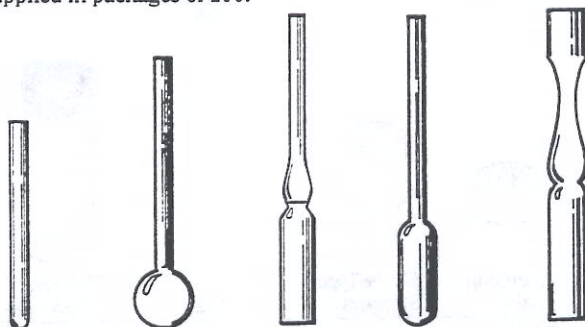
Ampules



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.



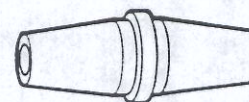
Size	Straight	Bulb-Type	Pre-Scored Flat Bottom	Round Bottom	Stoppering
1 ml	75727-10	75700-10	75706-10		
2 ml			75708-10		75723-10
4 ml	75728-10				
5 ml		75702-10	75710-10		
10 ml			75712-10	75716-10	
25 ml				75718-10	
50 ml				75720-10	

Accessories



Oxygen/Natural Gas Sealing Torch

75785 Torch specifically designed for flame sealing freeze dry ampules. Seals all types of heat-resistant glass. Shipping weight 3 lbs.



Adapter Valve

75934-01 Adapter connects ampules to $\frac{1}{2}$ " valves. Ten per package.



Stopper

75725-10 Stopper for stoppering ampules. 75723-10 100 per package.

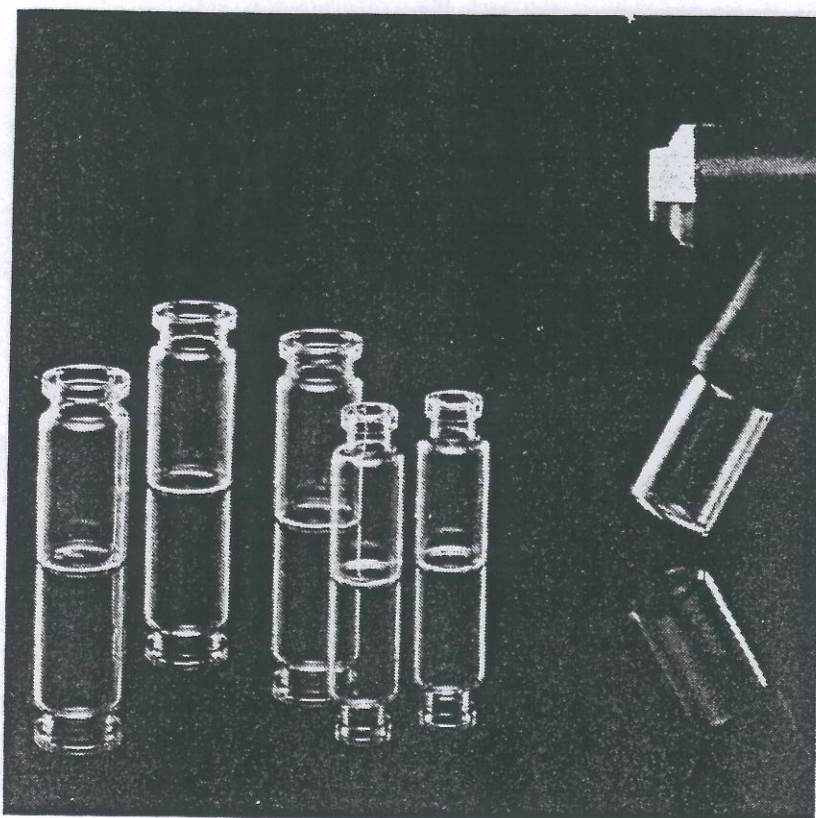


Trident Adapter

77627 3-way adapter permits attachment of 3 ampules to a single $\frac{1}{2}$ " valve. Ampules are attached using $\frac{1}{8}$ " surgical tubing (not provided). Cavity in adapter body for cotton filter media helps prevent contamination between samples.

ACCESSORIES

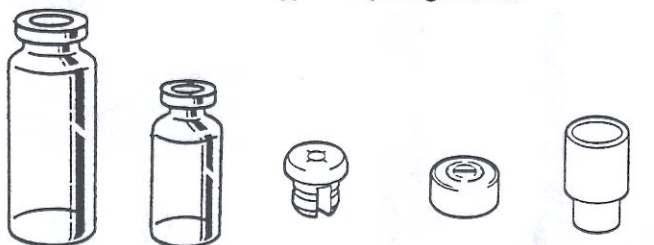
Serum Bottles and Vials



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

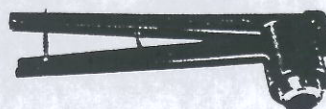
Serum Bottles

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		75750-10	75760-10	75770-10	
3 ml		75752-10	75760-10	75770-10	
5 ml	75730-10		75762-10	75771-10	75775-10
10 ml	75732-10		75762-10	75771-10	75775-10
20 ml	75734-10		75762-10	75771-10	75775-10
30 ml	75736-10		75762-10	75771-10	75775-10
50 ml	75738-10		75762-10	75771-10	75775-10
100 ml	75740-10		75762-10	75771-10	75775-10
125 ml	75742-10		75762-10	75771-10	75775-10

Accessories

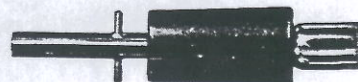


Seal Crimper

Seal crimper secures tear-away aluminum seals.

75780 Seal crimper for 13 mm corkage. Shipping weight 3 lbs.

75781 Seal crimper for 20 mm corkage. Shipping weight 3 lbs.

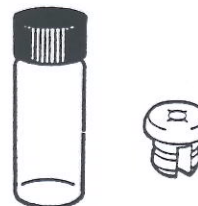


Vacuum Stoppering Adapter

75930 Adapter connects to valves for manual stoppering of 20 mm corkage serum bottles under original vacuum. Shipping weight 1 lb.

Threaded Vials

Stoppers and Threaded Vials with Screw Caps are supplied in packages of 200.



Size	Vials with Screw Caps	Stoppers
5 ml	77623	77622
10 ml	77626	77622

If you have any questions that are not addressed in this manual, or if you need technical assistance, please contact Labconco's Sales Information Department at either (800) 821-5525, and Service Information at (800)-522-7658 or (816) 333-8811, between the hours of 7:00 a.m. and 6:00 p.m. Central Standard Time.

Labconco's mailing address is:

Labconco Corporation
8811 Prospect Avenue
Kansas City, MO 64132

Visit Labconco through the Internet at:

<http://www.labconco.com>

or

email:labconco@labconco.com

Declaration of Conformity

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

Standard(s) to which conformity is declared: EN60950, 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.

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).

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

(Signature)

Date: _____
(Full Name)

(Position)

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