Lyph-Lock® Stoppering Tray Dryer

Freeze Dry System

Model 77560

INSTRUCTION MANUAL

1993

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Printed in U.S.A.

Introduction

Installation

Safety Precautions

Normal Operation

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Components Shipped

Carefully check the contents of the crate for damage that might have occurred in transit. Do not discard the crate 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 crate should contain the following:

Part Number	Description
77560-xx	Stoppering Tray Dryer
13364 or 13365	Power Cord 115 V Power Cord 230 V
18807-20	Screw (4)
19065-21	Nut (4)
19114-16	Washer (8)
19100-16	Lock Washer (4)
78479	Instruction Manual

General Description

The Labconco Lyph-Lock® Stoppering Tray Dryer is designed for laboratory lyophilization procedures. Its three shelves, with 600 square inches of area, are ideally suited for either batch samples or bulk product processing. A microprocessor precisely controls the temperature of the heat transfer fluid that circulates throughout the channels within the shelves. In addition, samples may be automatically cooled or heated in a pre-programmed sequence to comply with the researcher's unique protocol.

Samples may be prepared for lyophilization by pre-freezing them directly on the shelves of the Tray Dryer. After lyophilization, serum bottles, vials, or ampules may be stoppered under original vacuum on any of the three shelves using the unique pneumatic system. Shown below is a Stoppering Tray Dryer.

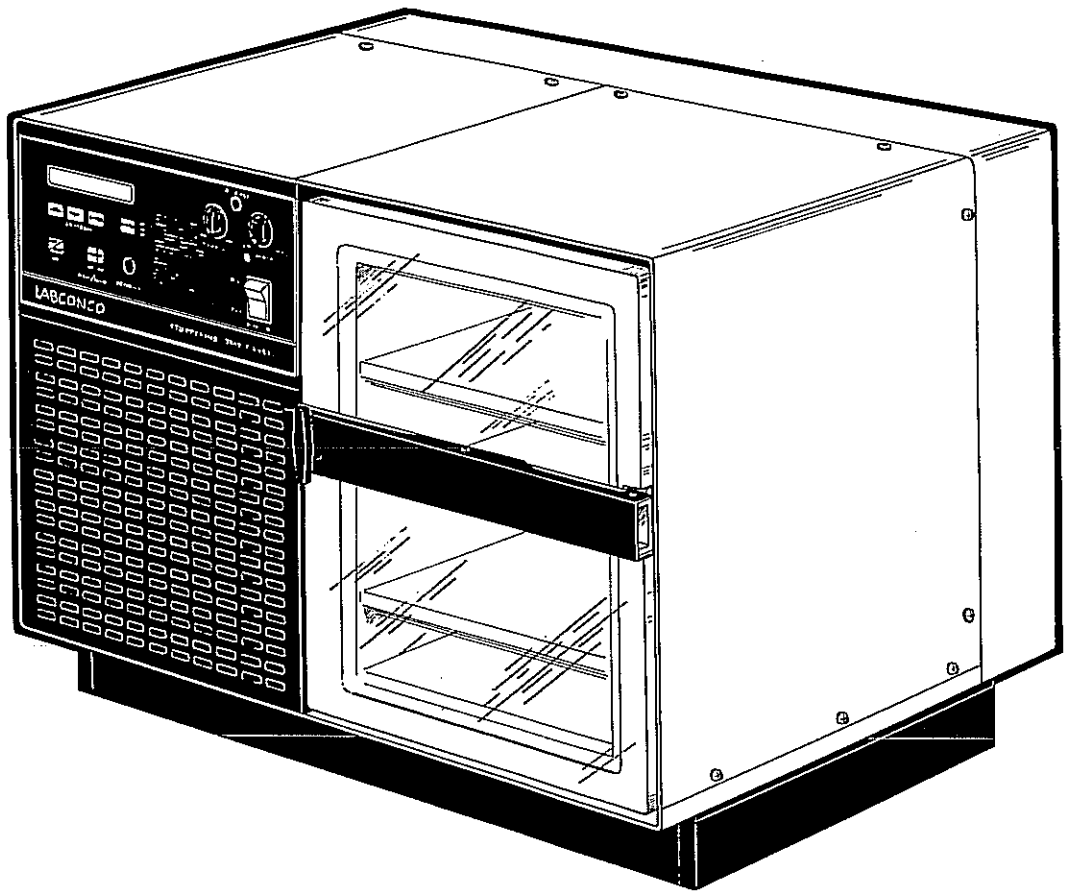


Figure 1

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

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. 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.

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 lowering the shelf temperature 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.

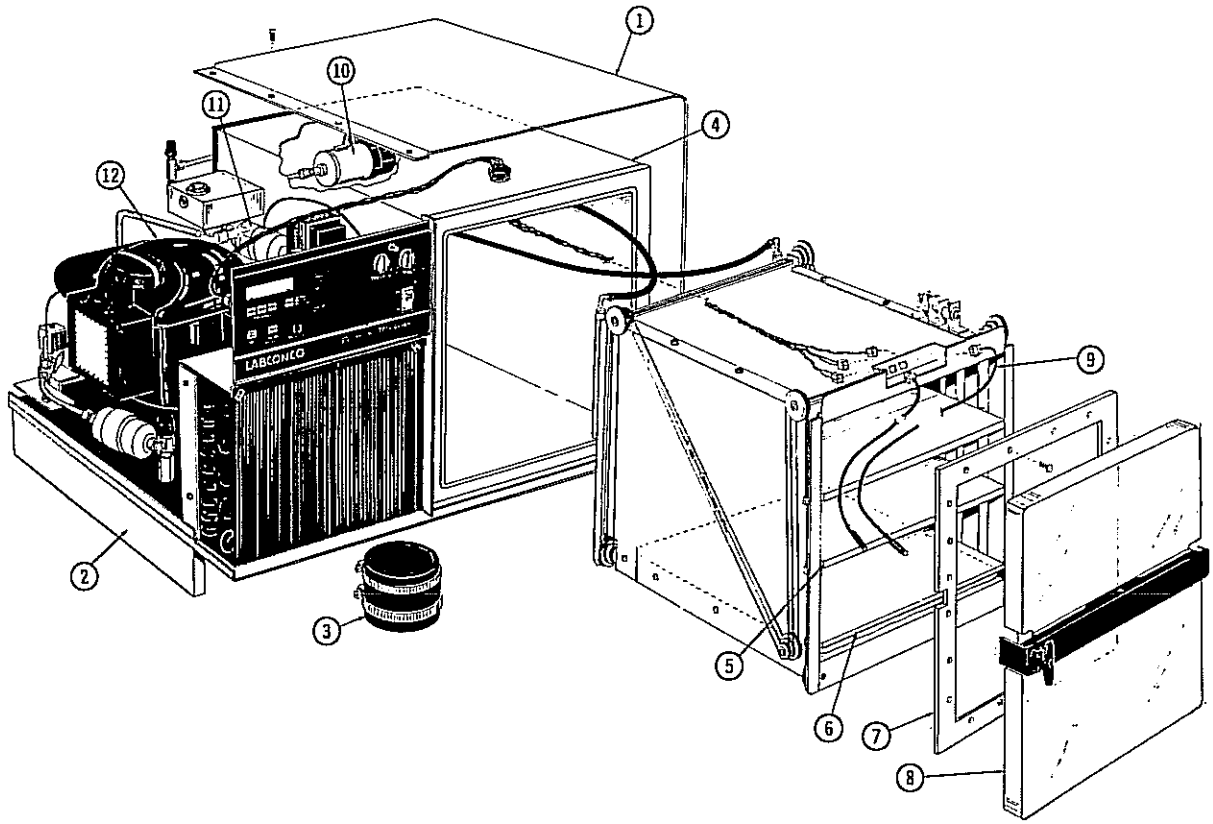


Figure 2

Component Identification (See Figure 2)

(1) **Cabinet.** The cabinet is epoxy powder coated steel for strength and durability.

(2) **Stand.** The stand mounts the Stoppering Tray Dryer to the Base Unit.

(3) **Vacuum Coupling.** A neoprene hose connects the Stoppering Tray Dryer to the condenser and vacuum system of the Base Unit.

(4) **Vacuum Chamber.** The welded chamber is constructed from heavy gauge stainless steel for high strength and corrosion resistance.

(5) **Sample Shelf.** Three temperature controlled processing shelves are provided.

(6) **Stoppering Mechanism.** The built in pneumatically activated mechanism moves the 3 shelves to provide positive stoppering of sample vials.

(7) **Door Gasket.** The neoprene gasket forms a leak tight seal between the vacuum chamber and the door.

(8) **Chamber Door.** The door is clear acrylic to provide good visibility into the chamber.

(9) **Sample Temperature Probe.** Three probes are supplied to monitor the temperature of samples.

(10) **Heater.** A high capacity heater is used to modulate the temperature of the heat transfer fluid that circulates throughout the shelves.

(11) **Circulation Pump.** A pump is used to circulate heat transfer fluid through the shelves.

(12) **Refrigeration Module.** A capillary type refrigeration system is used to cool the heat transfer fluid that circulates through the shelves.

Preparation

Lyph-Lock Stoppering Tray Dryers are designed to operate with the following Lyph-Lock Freeze Dry Systems.

6 Liter Benchtop	Model 77520-xx
6 Liter Console	Model 77530-xx & 77535-xx
12 Liter Console	Model 77540-xx & 77545-xx
12 Liter Console with Cascade Refrigeration System	Model 77610-xx
18 Liter Console	Model 77550-xx & 77555-xx

These systems must first be prepared for operation as explained in their Instruction Manuals.

Assembly (See Figure 3)

Remove the 4 plastic hole plugs from the work surface of the Freeze Dry System on which the Tray Dryer is to be mounted.

Make sure the lower clamp provided on the vacuum coupling opposite the end connected to the Tray Dryer chamber is loose. Secure this clamp with tape while mounting the Tray Dryer.

Lift the Tray Dryer into place on your Freeze Dry System while fitting the vacuum coupling over the manifold port and aligning the four mounting holes in the Tray Dryer support stand with the four holes in the Freeze Dry System's work surface.

Install the four bolts, nuts, lock washers, and washers provided (in a bag in the Tray Dryer's chamber). Remove the tape securing the lower clamp on the vacuum coupling and slide down over manifold connecting port. Orient the clamp screw for easy access and tighten the clamp to provide a leak-free connection.

The Tray Dryer is now installed and must be tested to make certain the system is free of leaks. To test, turn on the base unit refrigeration and allow the temperature to reach -40°C or lower. Close the door of the Tray Dryer and make sure the Stoppering Control is in the "OFF" position and the Vac Break control is in the "CLOSED" position. Start the vacuum pump and monitor the vacuum gauge. The vacuum on the base unit should reach 13×10^{-3}mBar or lower to confirm no leaks.

If 13 x 10^{-3}mBar cannot be achieved, consult the troubleshooting section of this manual and of the manual supplied with the Freeze Dry System.

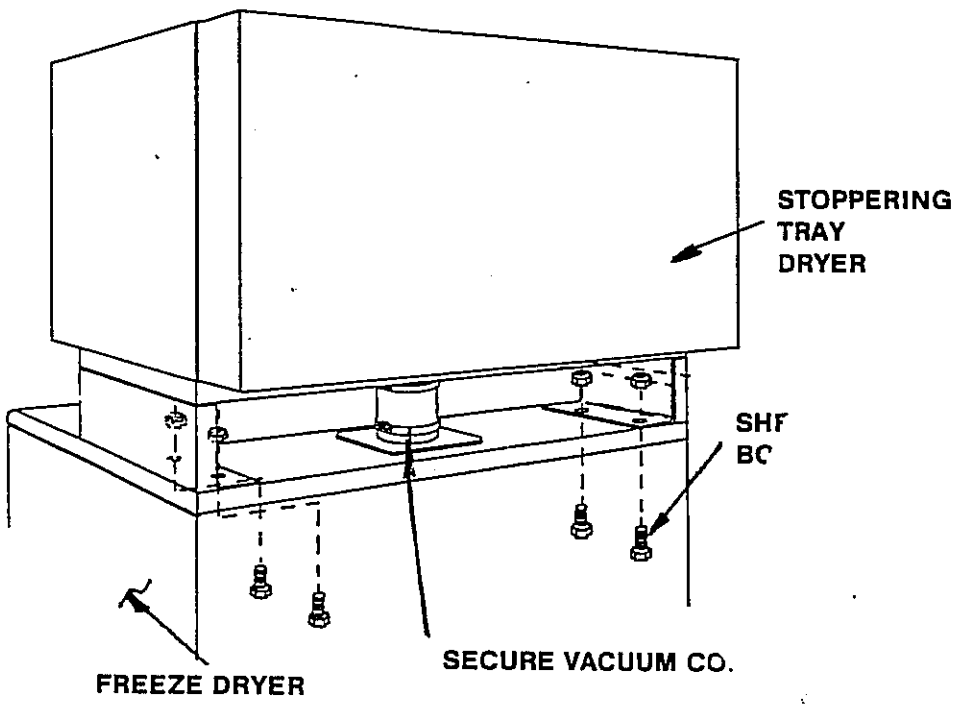


Figure 3

Utility Connections

Models 77560 & 77560-02 should be plugged into an electrical outlet rated at 115 VAC, 60 Hz, single phase, 20 amps. Model 77560-01 should be plugged into an electrical outlet rated at 230 V, 50 Hz, single phase, 10 amps.

Location

The freeze dry system should be located in an area that provides an unobstructed flow of air around the cabinet. This air cools the refrigeration system. The refrigeration system draws air through the grill on the front panel and exhausts it through the back of the cabinet. A minimum of 3" must be allowed between the back of the freeze dry system and the adjacent wall surface. Restriction of airflow into the system during operation could adversely affect performance.

General Precautions

Never attempt to perform any maintenance work without first disconnecting the Stoppering Tray Dryer from its power supply.

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 the collector coil of the base unit as serious damage to the refrigeration module may result. The release of refrigerant could injure your eyes.

Heat Transfer Fluid Precautions

The fluid's name and manufacture are:

Name: Lexsol 408M
Mfg: Santa Barbara Chemical Co.
927 Indio Muerto
Santa Barbara, CA 93140
805-963-7793

WARNING: The fluid used for heat transfer in this system is combustible and hazardous. Leaks and spills should be attended to immediately.

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

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

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

First Aid Procedures - Heat Transfer Fluid:

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

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

Inhalation: Remove patient to fresh air. If breathing stops, give artificial respiration, preferable mouth-to-mouth. Oxygen may be used if available. Get medical attention immediately, if required.

Ingestion: Get medical attention immediately.

Operation Checklist

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

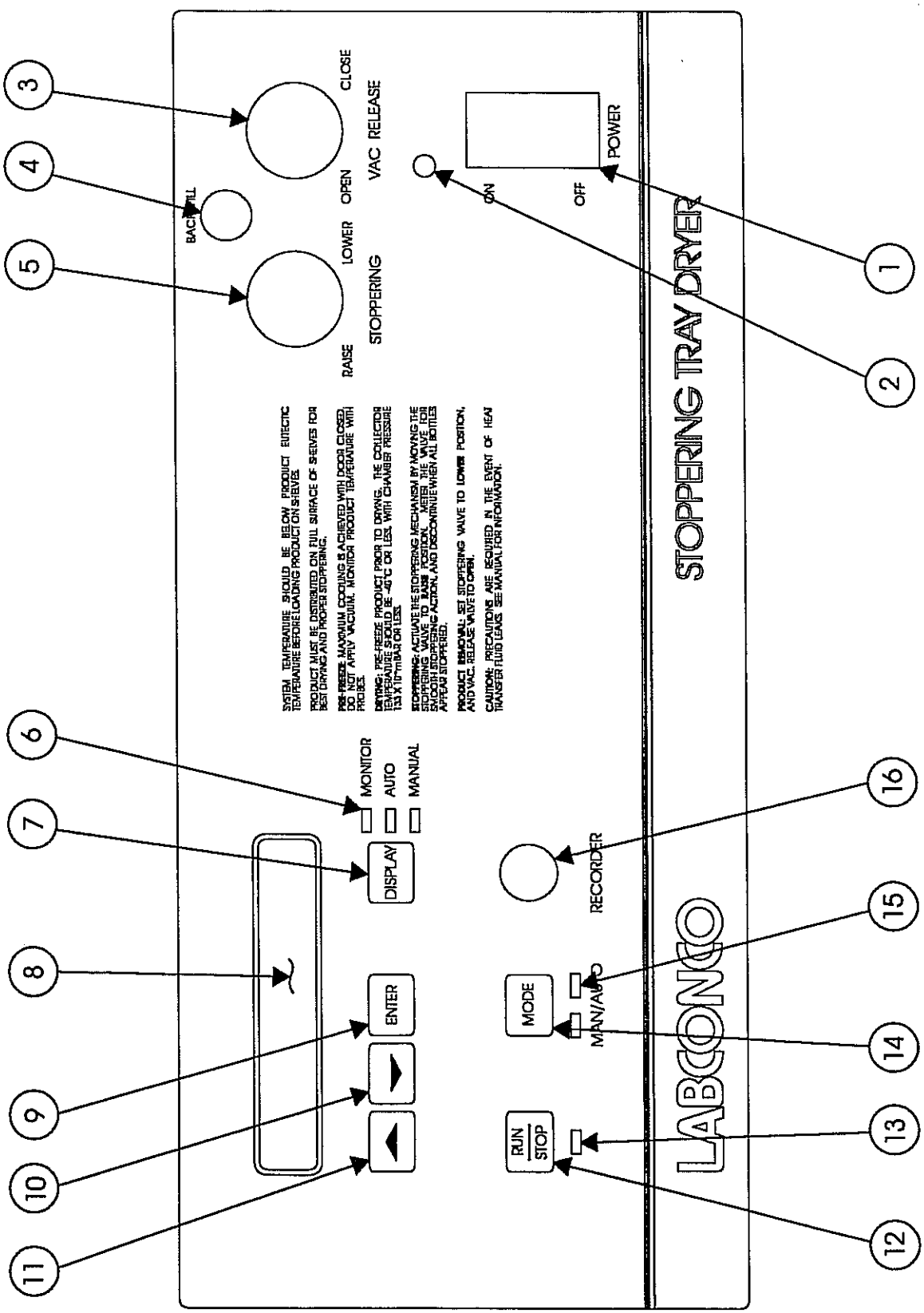
(1) Wipe the interior of the condenser chamber with a soft cloth or paper towel to remove any accumulated moisture.

(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 and the Stoppering Tray Dryer door gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the gaskets to obtain a proper vacuum seal.

(5) If the system contains the accessory support stand with valves, inspect each sample valve 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.

(6) Make sure that the Stoppering Control is in the "OFF" position and the Vac Break control is in the "CLOSED" position.


STOPPERING TRAY DRYER
LABCONCO
POWER
OFF
ON
VAC RELEASE
CLOSE
OPEN
LOWER
STOPPERING
RAISE
BACKFILL
MONITOR
AUTO
MANUAL
DISPLAY
ENTER
MODE
MAN/AUTO
RUN
STOP
RECORDER
1
2
3
4
5
6
7
8
9
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11
12
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15
16

Figure 4

Control Panel Identification (See Figure 4)

(1) **Power Switch.** Turns all power to Tray Dryer on or off.

(2) **Power Indicator.** Burns green steadily when power is on. If a power failure occurs during a run, the green indicator will flash when power is restored.

(3) **Vacuum Release.** Vents the chamber so the chamber door can be opened. This control can also vent gas into the chamber when the gas is properly connected to the backfill port.

(4) **Back Fill.** A regulated tank of gas may be connected to the port to allow the introduction of gas into the chamber during freeze drying. The port accepts 1/8" tubing.

NOTE: Ethylene oxide is not recommended for use in this Tray Dryer for decontamination because of its hazardous and corrosive properties.

(5) **Stoppering.** Controls the stoppering up and down movement. The mechanism will function only while the chamber is under vacuum. The stoppering action should be closely monitored when operating this control.

(6) **Display Indicators.** Amber lamps indicated which display "screen" is shown.

(7) **Display Selector.** Changes format of display from "monitor" to "auto" to "manual" screens.

(8) **Display.** Displays all necessary programming and operational data. Provides prompts to aid in programming.

(9) **Enter Switch.** Used in programming to enter a selected set point into memory.

(10) **Decrease Switch.** Used in programming to decrease a parameter set point.

(11) **Increase Switch.** Used in programming to increase a parameter set point.

(12) **Run/Stop Switch.** Initiates the start or stop of the lyophilization process.

(13) **Run/Stop Indicator.** Green lamp burns steady while freeze drying is in progress and turns off if either the programmed cycle is completed or the process is terminated in mid cycle. The green lamp flashes if freeze drying is in progress and an out-of-specification condition exists.

Component Identification (Con't)

(14) **Mode Selector Switch.** Selects the mode of operation - either manual or automatic.

(15) **Mode Indicator.** Amber lamp indicates whether the control is set to operate either in the automatic or manual mode.

(16) **Recorder Jack.** A DIN, 7 pin, 240° receptacle permits the attachment of a recorder so system temperature and vacuum as well as three sample temperatures and the program segment may be recorded.

Manual Control Operation

(1) Turn the Power switch On. The green indicator will light and the display will become active.

(2) Push the Display button until the Manual LED lights. This will cause the manual display screen to be shown with the previously entered set point temperature.

SET POINT TEMP - 30°C

(3) If the set point needs to be changed, press the Increase or Decrease button until the desired set point temperature is displayed.

(4) Press Enter. If Enter is not pressed within 10 seconds, the set point will revert to the previously entered set point temperature.

(5) Press Mode button until the Manual LED lights.

(6) Press Run/Stop button. The amber LED under the button will light and the refrigeration system and/or heater will operate.

(7) To change the set point temperature in the middle of a run, repeat steps 2, 3 and 4. The system will adjust to the new temperature set point.

(8) To monitor system operation, press Display until the Monitor LED lights. The display will simultaneously show system temperature, vacuum and 3 sample temperatures if sensors were plugged into their jacks at the front of the chamber and inserted into sample vials. If sensors are not plugged into their jacks, the probe temperature display will show "LIM."

The segment number will always indicate "1" while operating in the manual mode. Segment numbers are used only in the automatic mode to identify portions of the programmed cycle. Temperatures are displayed as °C and vacuum is displayed as 10^{-3}mBar. Above 6000 x 10^{-3}mBar the display will show "HI" vacuum. System temperature rather than probe temperature is the parameter that is regulated by the controller.

PROBE 1 = -30	2 = -30	3 = -30
TEMP = -30	SEG = 1	VAC = 15

(9) To stop, Push Run/Stop button, which will turn off the amber LED under the switch, the refrigeration system and the heater. To turn off the entire system, turn the Power Switch off.

Automatic Control Operation

Description

The Stoppering Tray Dryer is equipped with a microprocessor based controller that permits temperature to be programmed using as many as 5 different segments. Each segment consists of a temperature ramp function and a temperature hold function. The ramp allows the temperature of the sample to be increased or decreased at any desired rate within the capacity of the heating and cooling systems of the Stoppering Tray Dryer.

A Stoppering Tray dryer without samples on shelves is capable of cooling at a rate of approximately .5°C/minute and can heat at approximately 1.5°C/minute.

When the desired temperature is achieved, the hold function will maintain that temperature for the programmed length of time. The microprocessor control has a built in memory of the last entered program to allow the identical protocol to be repeated by simply pushing the Run button. A typical 3 segment program is shown in Figure 5.

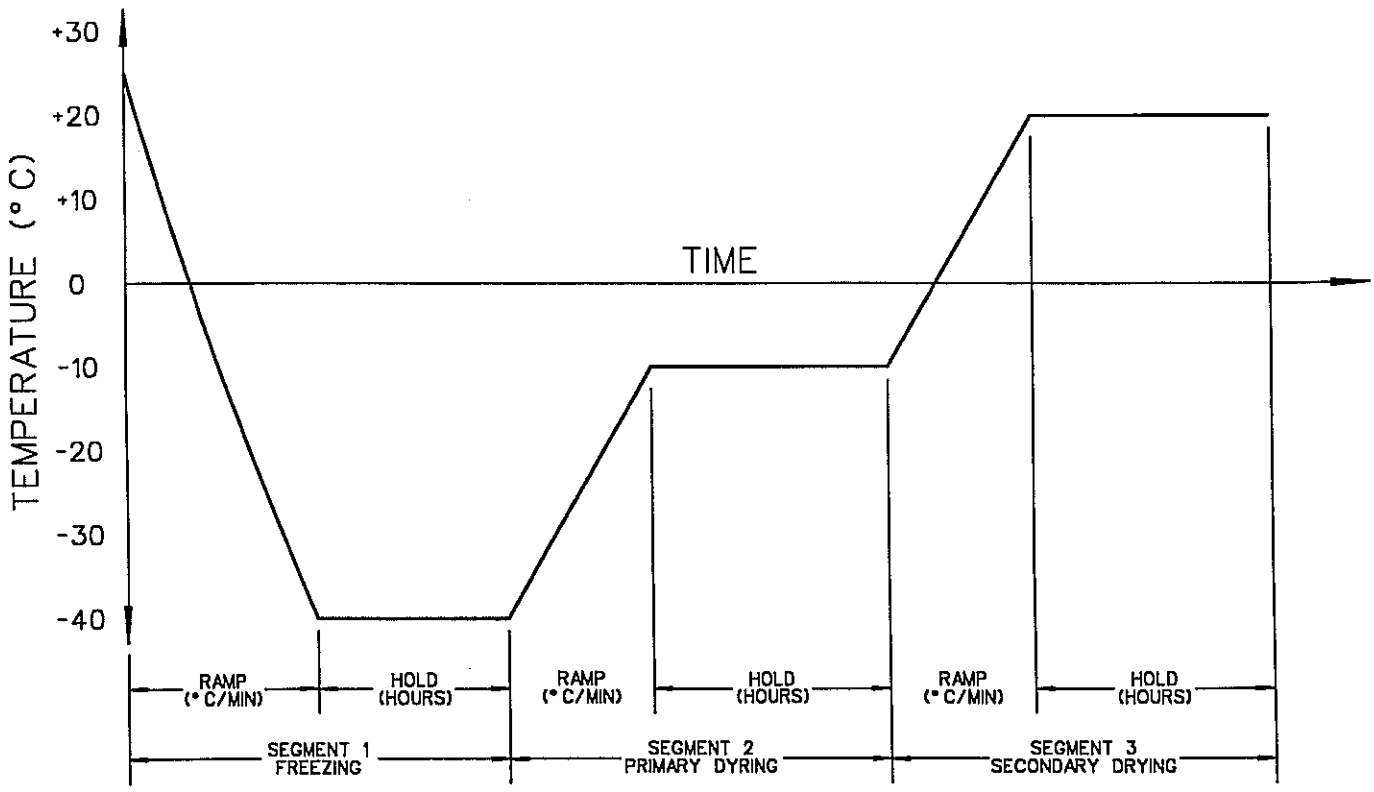


Figure 5

Programming

(1) Turn the Power switch On and the green indicator will light and the display will become active.

(2) Push the Display button until the Auto LED lights. This will cause the Auto Display screen to be shown with the previously entered Segment 1 parameters displayed. The letter "S" in Segment will flash to indicate that this parameter is ready to be programmed.

SEGMENT 1	RAMP 0.45°C/MN
HOLD - 30°C	TIME 15.5 HR

(3) If all displayed parameters for Segment 1 are as they should be, press the program Increase button. Since the letter "S" in Segment was flashing, the displayed number "1" will change to "2".

(4) If all displayed parameters for Segment 1 are not as they should be, press Enter. This will cause the "S" in "Segment" to stop flashing and the "R" in Ramp to flash.

(5) The temperature ramp speed can now be programmed. Press the program Increase or Decrease button until the desired ramp is displayed. Press Enter. "R" will stop flashing and the letter "H" in Hold will flash.

(6) The hold temperature is now ready to be programmed. Press the program Increase or Decrease button until the desired hold temperature is displayed. Press Enter. "H" will stop flashing and the letter "T" in Time will flash.

(7) The hold time is now ready to be programmed. Press the program Increase or Decrease until the desired hold time is displayed. Press Enter. "T" will stop flashing and "S" in Segment will flash again.

(8) If all displayed parameters for Segment 1 are not as they should be, repeat steps 4 thru 7.

(9) If all displayed parameters for Segment 1 are as they should be, press Program Increase button to advance the display from Segment "1" to "2". Segment "2" can now be programmed following the previously described procedure.

Programming Rules

(1) When the segment number is changed, previously programmed set point parameters for that segment for ramp, hold and time will be displayed simultaneously with the number change.

(2) If Enter is not pushed within 10 seconds of changing a set point, the display will revert back to the last previously entered value. Enter must be pushed to insert a parameter into memory.

(3) If the complete cycle does not require 5 segments:

(a) Program the desired number of segments.
(b) Roll the display to the next higher segment number.
(c) Enter Ramp "0.00"
Hold "Same as last running segment temperature"
Time "00.0"
(d) Repeat (b) and (c) if required for all remaining segments.
All 5 segments must be programmed.

(4) The system prompts which parameter is ready to program by flashing the first letter of key words one at a time in the following sequences:

Segment, Ramp, Hold, Time.

(5) If it is desired to change from the Manual mode to the Auto mode, or vice versa, the Run/Stop switch must be off prior to changing modes.

Starting The Freeze Dry Cycle

After all 5 segments have been programmed, push the Run/Stop button. The amber LED under the button will light and the refrigeration system and/or the heater will operate.

Changing The Program Parameters In The Middle Of A Run

(1) Press Display to obtain Monitor screen. Note current operating segment.

(2) Press Display to obtain Auto screen.

(3) Enter a segment number that will occur after the current segment that is running. The presently running segment cannot be modified while the system is running.

(4) Modify as required.

(5) Press Enter.

Stopping The Freeze Dry Cycle

(1) At the end of the last programmed segment the freeze dry system will automatically stop. The refrigeration system and the heater will shut off. The display will show Segment "E" (End).

(2) To stop before the completion of the last programmed segment, press Run/Stop. The amber lamp will turn off as will the refrigeration system and heater. The display will remain active but time functions will stop operating.

(3) The entire system can be shut off by pressing the Power switch.

(4) If the Power switch is shut off prior to stopping the cycle with the Run/Stop switch, when the Power switch is again turned on, the system will resume operation and attempt to complete the programmed cycle from the point at which it was shut off.

Monitoring The System Operation

Press the Display button until the monitor LED lights. The display will show system temperature, system vacuum, and 3 sample temperatures if sensors were plugged into their jacks at the front of the chamber and inserted into sample vials. If sensors are not plugged into their jacks, the probe temperature display will show "LIM". The temperatures are displayed as °C while the vacuum is displayed 10^{-3}mBar. Above 6000 x 10^{-3}mBar the display will show "HI" vacuum.

While operating in the Auto Mode, the display will indicate what segment of the program is presently controlling the process. The display will show the segment number followed by an "R" or "H" indicating whether the temperature is Ramping or Holding.

PROBE 1 = -30	2 = -30	3 = -30
TEMP = -30	SEG = 1H	VAC = 15

System temperature rather than probe temperature is the parameter regulated by the controller.

Shelf Spacing Adjustment

Prior to loading the shelves with the product to be freeze dried, adjust the shelves to the 3-shelf, 2-shelf or 1-shelf position. It is best to observe the shelf supporting mechanism and practice while reading these procedures. In the 3-shelf position, the spacing between the shelves will accommodate a serum bottle with the stopper raised up to a maximum height of 75 mm (2.9 inches). Minimum serum bottle height with lowered stopper for bottles on 3 shelves is 38mm (1.5 inches).

For a 2-shelf system, grasp the top shelf and raise it until it latches in place. Then raise the middle shelf approximately 1 inch until it latches in a position to allow a two equally-spaced shelf system. The spacing between shelves will accommodate a bottle with the stopper raised up to a maximum height of 107 mm (4.2 in.). Minimum serum bottle height with lowered stopper for a 2-shelf system is 56mm (2.2 in.)

For a single-shelf system, raise both the top and middle shelves until they latch in place in their top positions. The spacing for a single-shelf system will accommodate a bottle with raised stopper up to a maximum height of 196mm (7.7 in.), and the minimum height of a bottle with lowered stopper for successful stoppering is 114mm (4.5 in.).

To unlatch the shelves, press the latch protruding from the side of the shelf support structure and move the latch until it clears the shelf and allows the shelf to drop. The top shelf has one latched position, the middle shelf has two latched positions, and the bottom shelf should not latch in any position.

Shelf Loading

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

Your Stoppering Tray Dryer has a unique system for keeping the shelves level and moving smoothly while stoppering. If a problem occurs during stoppering, the system is designed to flex a small amount to lessen the possibility of damage to the unit.

The stoppering action should be monitored by looking through the chamber door. Cease stoppering when all bottles appear stoppered or if a problem occurs. Distributing the serum bottles evenly on the shelves also aids in consistent drying of all serum bottles on each shelf.

Sample Freezing

Before the freeze dry process can occur, the product to be dried must be in a frozen state. This can be accomplished in a freezer separate from the Stoppering Tray Dryer or on the shelves in the Stoppering Tray Dryer. First the shelves should be adjusted to accept the size of the selected sample containers. Next, turn On the power switch and set the controls to cool the shelves. This is most simply done by setting the temperature in the manual mode as described on page 19 although segment 1 in the automatic mode can also be programmed to cool the shelves. Close the chamber door. Load the samples onto the shelves after the system temperature is below the sample eutectic temperature. If desired, place one temperature probe in a sample vial on each shelf. The temperature can be monitored by pressing the Display button until the Monitor LED lights.

Freeze Drying

After the product is frozen, turn on the Lyph-Lock Freeze Dry System base unit. Following the instructions for the unit, this may be accomplished in either the automatic or manual mode.

When the collector temperature is less than -40°C and the vacuum is less than 133×10^{-3}mBar, the manual mode set point temperature may be adjusted or the automatic mode program may be initiated. At no time during the primary drying phase should the product temperature be allowed to rise higher than the eutectic temperature.

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

Stoppering

The stoppering operation (when desired) is performed after the freeze dry process is complete. To stopper, move the Stoppering control toward the "RAISE" position. This action allows the actuator beneath the bottom shelf to inflate causing the bottom shelf to rise. The bottles on the shelf will then contact the middle shelf causing it to rise and the top shelf will eventually be contacted and rise. When all three shelves have raised and made contact with each other, stoppers will be pressed into the vials. Monitor the stoppering process by looking through the chamber door.

When all of the bottles appear to be stoppered, move the Stoppering control to the "LOWER" position. This opens the diaphragm to the vacuum pump, which deflates the diaphragm. Opening the vacuum release control also deflates the stoppering diaphragm. The Stoppering control should be left in the "LOWER" position when not stoppering.

Vacuum Break/Backfilling

To open the chamber door, the vacuum must be released. To release the vacuum, move the Vacuum Release control to the "OPEN" position and shut off the vacuum pump using the switch on the base unit.

Air enters the vacuum chamber through the Back Fill port. When the sound of air through the Back Fill port is no longer audible, the chamber door is ready to open. To backfill the chamber with a gas, connect a cylinder of the desired gas to the Back Fill port. The port will accept 1/8" tubing. The bottle must be equipped with a regulator set to 15 psi maximum. The gas can be metered with the Vacuum Release control and chamber pressure monitored with the freeze dry system's vacuum gauge.

Recorder Jack

The system temperature, 3 sample temperatures, vacuum level and segment number can be recorded during operation by connecting a recorder to the recorder jack on the control panel. The recorder jack is a DIN receptacle 7 pin 240°. The pin configuration and output is as follows:

Pin 1 - Ground

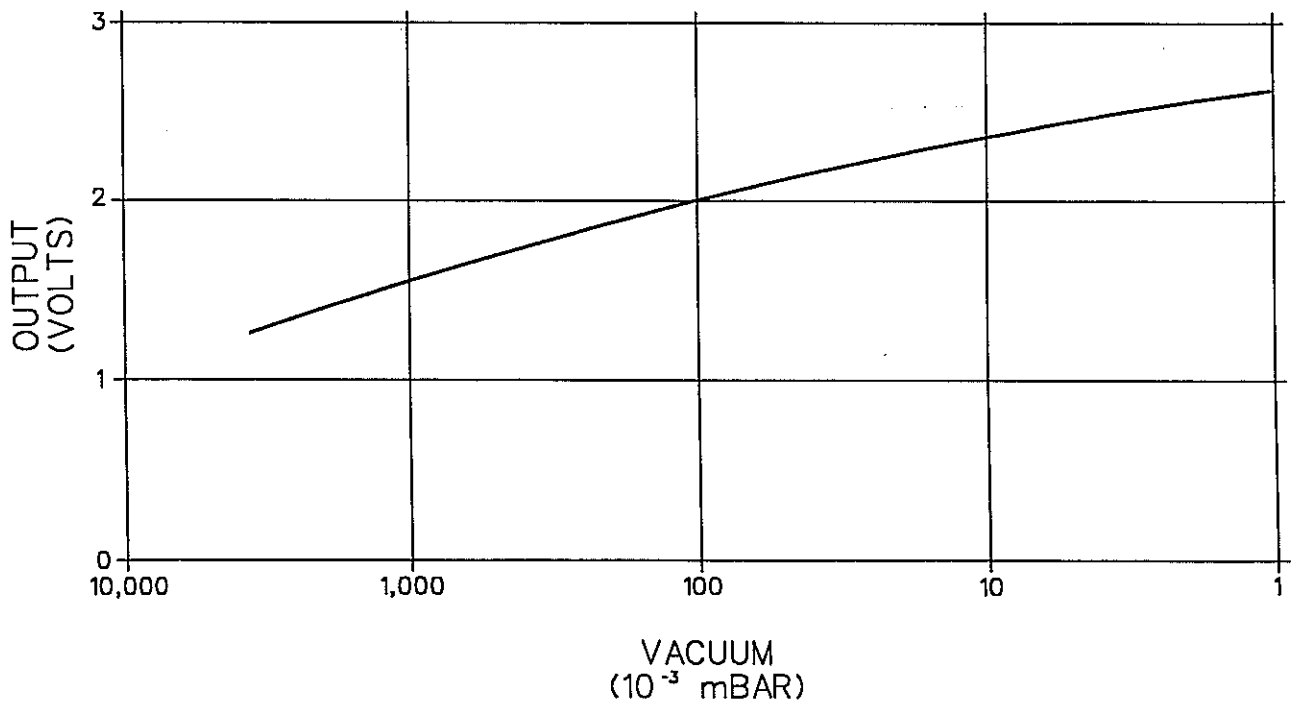
Pin 2 - System Temperature - Output
40°C = 4.600 VDC
0°C = 4.016 VDC
-40°C = 3.432 VDC

Pin 3 - Sample Temperature #1 - Output
40°C = 4.600 VDC
0°C = 4.016 VDC
-40°C = 3.432 VDC

Pin 4 - Sample Temperature #2 - Output
40°C = 4.600 VDC
0°C = 4.016 VDC
-40°C = 3.432 VDC

Pin 5 - Sample Temperature #3 - Output
40°C = 4.600 VDC
0°C = 4.016 VDC
-40°C = 3.432 VDC

Pin 6 - Vacuum - This graph shows the approximate D.C. voltage output versus vacuum reading.



Pin 7 - Segment Number - Output	Prior to Start	=	O V DC
	Seg 1 Ramp	=	.50
	Seg 1 Hold	=	.75
	Seg 2 Ramp	=	1.00
	Seg 2 Hold	=	1.25
	Seg 3 Ramp	=	1.50
	Seg 3 Hold	=	1.75
	Seg 4 Ramp	=	2.00
	Seg 4 Hold	=	2.25
	Seg 5 Ramp	=	2.50
	Seg 5 Hold	=	2.75
	Cycle Complete	=	3.00

Warning System

Power Failure

If a power failure occurs while a run is in progress, when the power is restored, the green Power indicator will flash. Once power is restored, the process will continue as programmed until completion. Turning the power switch Off cancels the flashing warning.

Temperature

If the manual set point temperature or automatic hold temperature varies more than $\pm$ 3°C as measured by the system temperature probe, the green Run indicator and the word "temp" on the display will flash until the end of the run.

Vacuum

Once the system vacuum has pulled down and stabilized at a point where it changes less than 5×10^{-3}mBar in 5 minutes, if the vacuum should decay more than 50×10^{-3}mBar, the green Run indicator and the word "vac" on the display will flash until the end of the run.

Under normal operation, your Freeze Dry System 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, leak-tight system or close the purge valve if the base unit is equipped with a purge valve. The gas ballast must be open.

Monthly

(1) The rubber components on the freeze dry system 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 chamber door.

(3) Using a soft cloth, sponge, or chamois and a mild, non-abrasive soap or detergent, clean the top, sides, 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) The refrigeration system condenser should be cleaned once every 6 months. This aids in maintaining the proper air flow which is essential to long life and peak performance of the Stoppering Tray Dryer. Access to the condenser requires removal of the two top screws which hold the front grille in place. The grille then is simply lifted free.

Cleaning on the refrigeration condenser face is best accomplished using a vacuum cleaner with brush attachment. More frequent cleaning is required if your unit is operated in a dirty environment.

(2) All monthly activities.

Decontamination

When freeze drying biological substances, it is often necessary to decontaminate the system. A surface decontaminate should be used to clean the accessible surfaces. The use of ethylene oxide is not recommended because of its hazardous and corrosive nature.

Under a no-load condition with the door closed and no vacuum, the shelves should reach -40°C in approximately 2 hours. This performance is based upon an ambient temperature of 24°C (75°F) at the rated voltage and 60 Hz electrical service.

If any repairs are required on the refrigeration module, contact your local laboratory supply dealer or Labconco Corporation at (800) 821-5525 or (816) 333-8811. Repairs should be made by an authorized Labconco service agency.

VACUUM LEAK DETECTION

Your freeze dry system can achieve an ultimate vacuum of 13×10^{-3} mBar or lower under a no-load condition. 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 system 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 manifold if so equipped 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 system 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. Refer to the vacuum pump manufacturer's instructions for further information.

(3) Check the collector chamber lid gasket and the Stoppering Tray Dryer door 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.

(6) The Stoppering Tray Dryer chamber and collector chamber must be dry.

If any repairs are required on your freeze dry system, contact your local laboratory supply dealer or Labconco Corporation at (800) 821-5525 or (816) 333-8811. Repairs should be made by an authorized Labconco service agency.

Access To Mechanical Components

(1) To gain access to the refrigeration system, pump, tank, and the controller and control panel components, it is necessary to remove the cover on the left side.

(2) First remove the ten screws which hold the cover in place. Lift off the cover and set it aside. The wiring diagram label is on the inside of this cover.

(3) Additional access is available by removing the remaining four screws which hold the rear panel in place and lifting it off the unit.

Shelf And Stoppering System Removal

(1) The shelf and stoppering system may be removed from the chamber as an assembly for repairs if needed.

(2) Remove the plexiglass door by taking out the two allen head screws located on the mounting frame of the cross bar.

(3) Remove the door gasket. Carefully pull loose all of the plastic fasteners (located around the periphery of the chamber) from the chamber. Leave the spacer blocks behind the gasket attached to the gasket.

(4) Place a support in front of the chamber and slide the shelf and stoppering system out approximately 2 inches.

(5) Loosen the clamp securing the stoppering diaphragm tube (located in the lower center at the back of the chamber) and remove the tube. Slide the assembly out of the chamber and onto the support.

(6) The assembly is now ready for repairs. Whenever possible, do not break the fluid system.

CAUTION: Read fluid precautions in this manual on page 13 and 14 when repairs concerning the fluid system are required.

(7) Reverse the removal procedures for installation of the shelf and stoppering system. Make sure the fluid inlet and outlet tubes are routed on top of the assembly.

Filling The Fluid System (See Figure 6)

(1) Make sure the Power switch is in the Off position.

(2) Remove the left cover. Unplug the refrigeration system from the junction block on the electrical wire harness (Item 3).

(3) Remove the insulation and both caps from the fluid circulation valve, which is located directly behind the fluid reservoir (Item 1).

(4) Attach a flexible tube to the exposed port (service port). The port is 3/8" x 45° flare (Item 4).

(5) Remove the fluid reservoir cover (Item 2) and place the opposite end of the tubing into the fluid reservoir.

(6) Fill the reservoir slowly allowing the fluid to drain through the system. When the system appears full, prepare to start the circulation pump.

(7) Open the fluid circulation valve by completely "down-seating" (turn top valve stem completely clockwise) to direct fluid to by-pass through tubing to fluid reservoir.

(8) Activate the pump by moving the Power switch to the On position. The fluid system will circulate with the fluid going through the add-on tubing, purging itself of air. Add fluid when needed to keep fluid in the reservoir. After the fluid stream is established, tilt the unit backward 1" for 2 minutes. Observe the fluid stream and repeat the tilt procedure if it is not free of bubbles.

(9) After the system is free of air "up-seat" fluid circulation valve (turn valve stem completely counter-clockwise, closing the fluid/tubing bypass).

(10) Turn off the system. Plug the refrigeration system into the junction block and turn On the system. Set the controller to operate at 24°C (75°F).

(11) When the system temperature stabilizes, add or remove fluid from the reservoir to the level indicated by the label.

(12) After filling the fluid system to the proper level, turn off the temperature control switch and allow unit to reach +30°C system temperature. Apply vacuum to the unit and when the system is 133 x 10^{-3}mBar or less, install the cap on the tank.

(13) Remove the tubing. Replace the caps on the valve and replace insulation and covers.

COMPONENT IDENTIFICATION ILLUSTRATION

1. Fluid Circulation Valve
2. Fluid Reservoir and Cap
3. Electrical Junction Block
4. Flexible Tubing

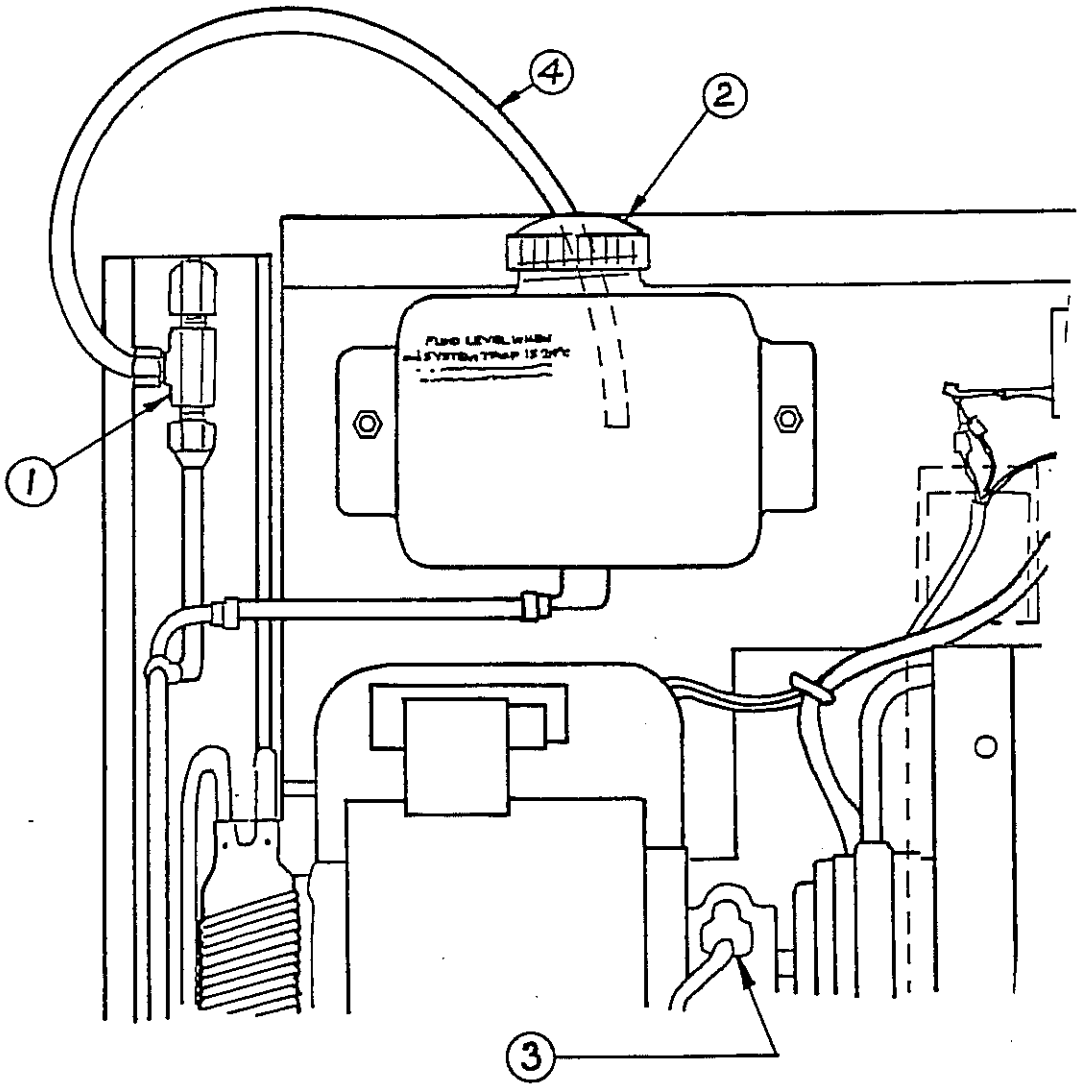
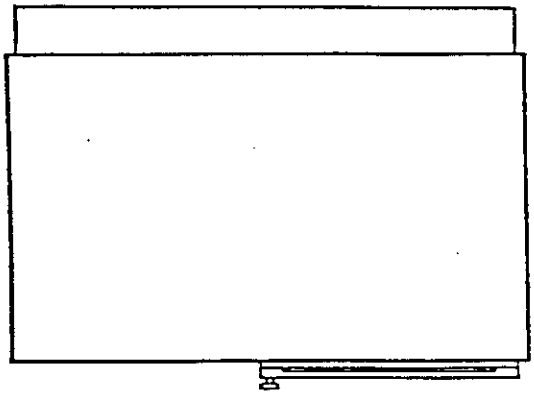


Figure 6



Serum Bottle Capacity of the Stoppering Tray Dryer

Size	Labconco No.	Shelf Capacity	No. of Shelves	Total Capacity
2 ml	75750	399	3	1197
3 ml	75752	296	3	888
5 ml	75730	255	3	765
5 ml	77623	255	3	765
10 ml	75732	195	3	585
10 ml	77626	255	3	765
20 ml	75734	132	3	396
30 ml	75736	86	3	258
50 ml	75738	72	2	144
100 ml	75740	42	2	84
125 ml	75742	36	1	36

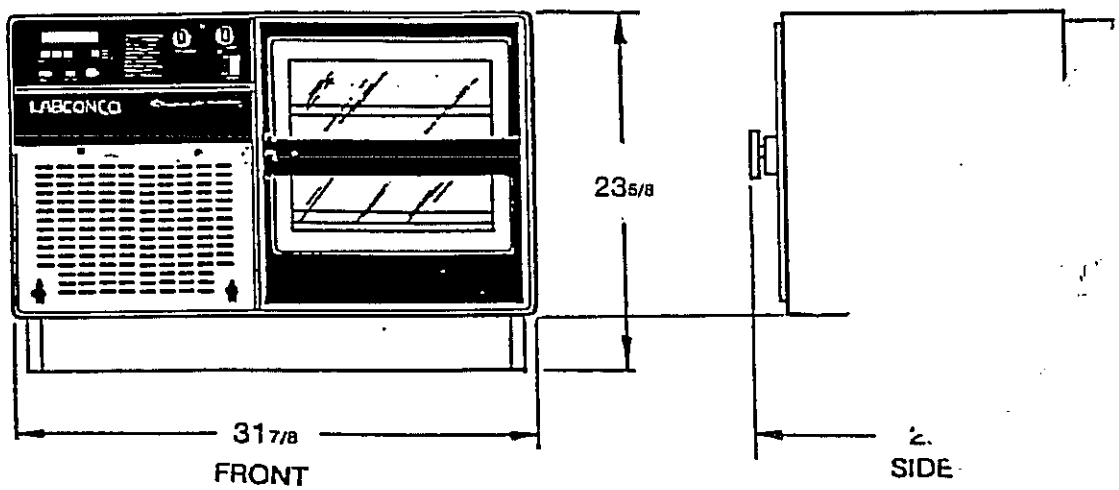


Figure 7

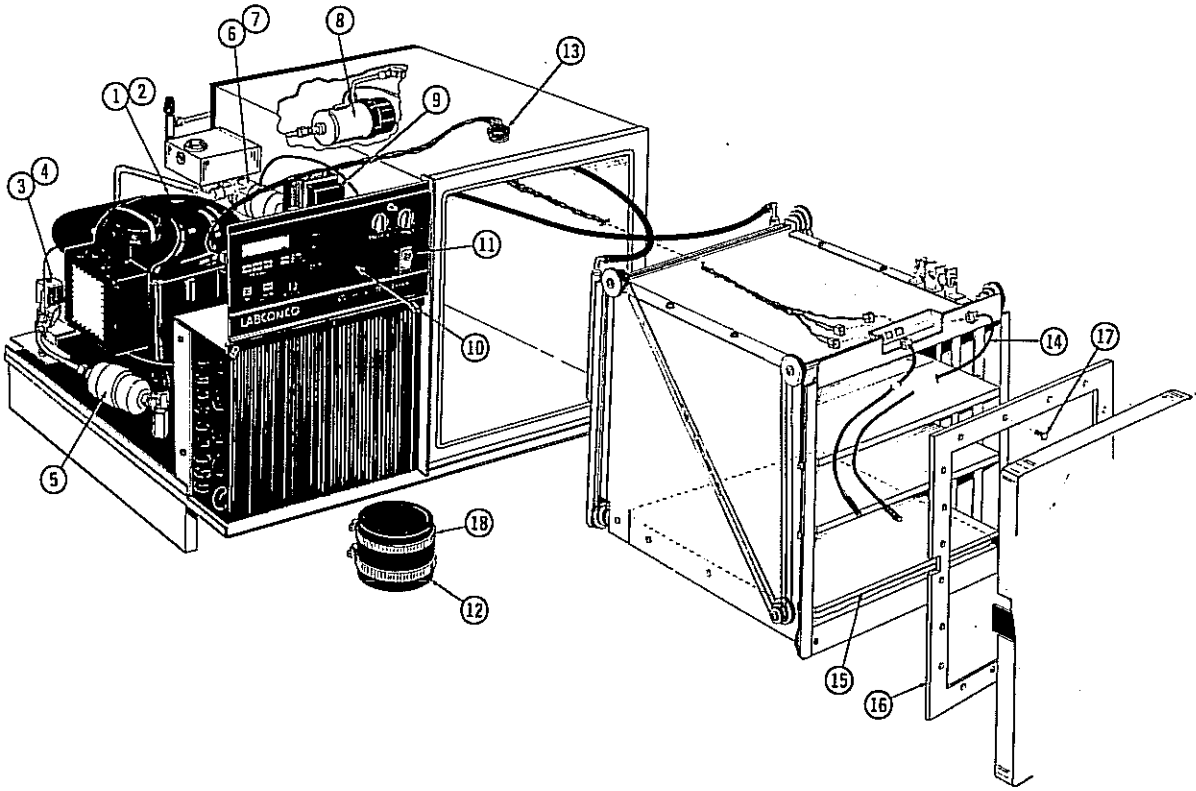


Figure 8

REFERENCE NUMBER	QUANTITY	CATALOG NO.	DESCRIPTION
1	1	77441	Refrigeration Unit (115 V)
1A	1	77441-02	Refrigeration Unit (230 V)
2	1 lb.	76222-01	Refrigerant
3	1	13603	Solenoid Valve
4	1	13604	Coil-Solenoid Valve (115 V)
4A	1	13604-01	Coil-Solenoid Valve (230 V)
5	1	14895	Dryer - Refrigeration
6	1	77874	Pump
7	1 gal.	77944	Heat Transfer Fluid
8	1	77813	Heater (115 V)
8A	1	77813-01	Heater (230 V)
9	1	77896	Transformer & Wire Harnes (230 V only)
10	1	78481	Controller Panel Assembly
11	1	13023	Switch
12	1	76352	Vacuum Coupling
13	1	78021	Bulkhead Harness
14	3	78416	Temperature Probe
15	1	77655	Diaphragm Assembly
16	1	77663	Chamber Gasket
17	20	19262	Clip - Gasket
18	2	19654	Hose Clamp

MODEL NUMBER	ELECTRICAL REQUIREMENTS
77560	115 V - 60 Hz 1 Phase - 18.8 Amp
77560-01	230 V - 50 Hz 1 Phase - 9.3 Amp
77560-02	115 V - 60 Hz 1 Phase - 18.8 Amp

All ratings include only the Stoppering Tray Dryer without auxiliary equipment.

Models 77560-00 and 77560-02
(115 VAC, 60 Hz, 1 Phase)

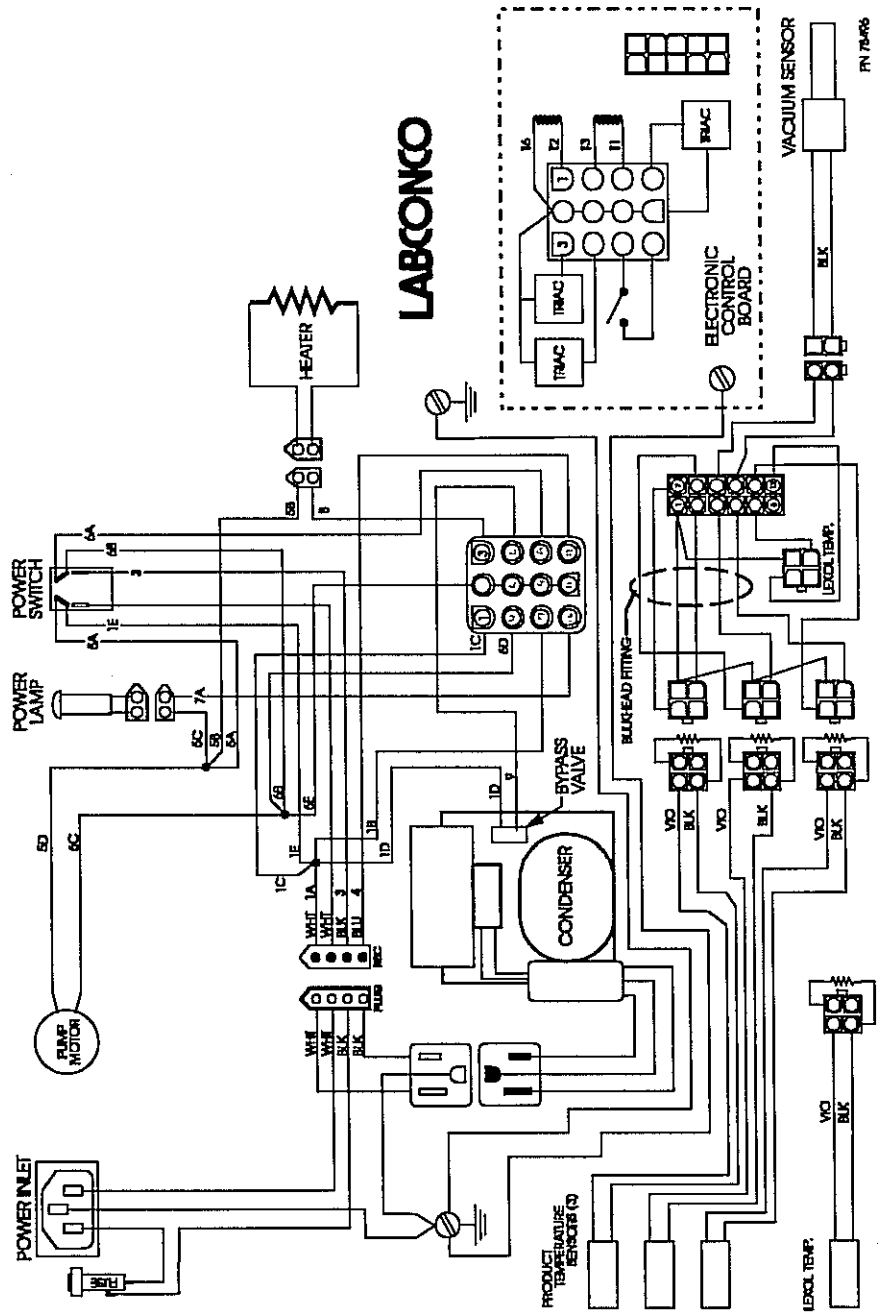


Figure 9

Model 77560-01
(230 VAC, 50 Hz, 1 Phase)

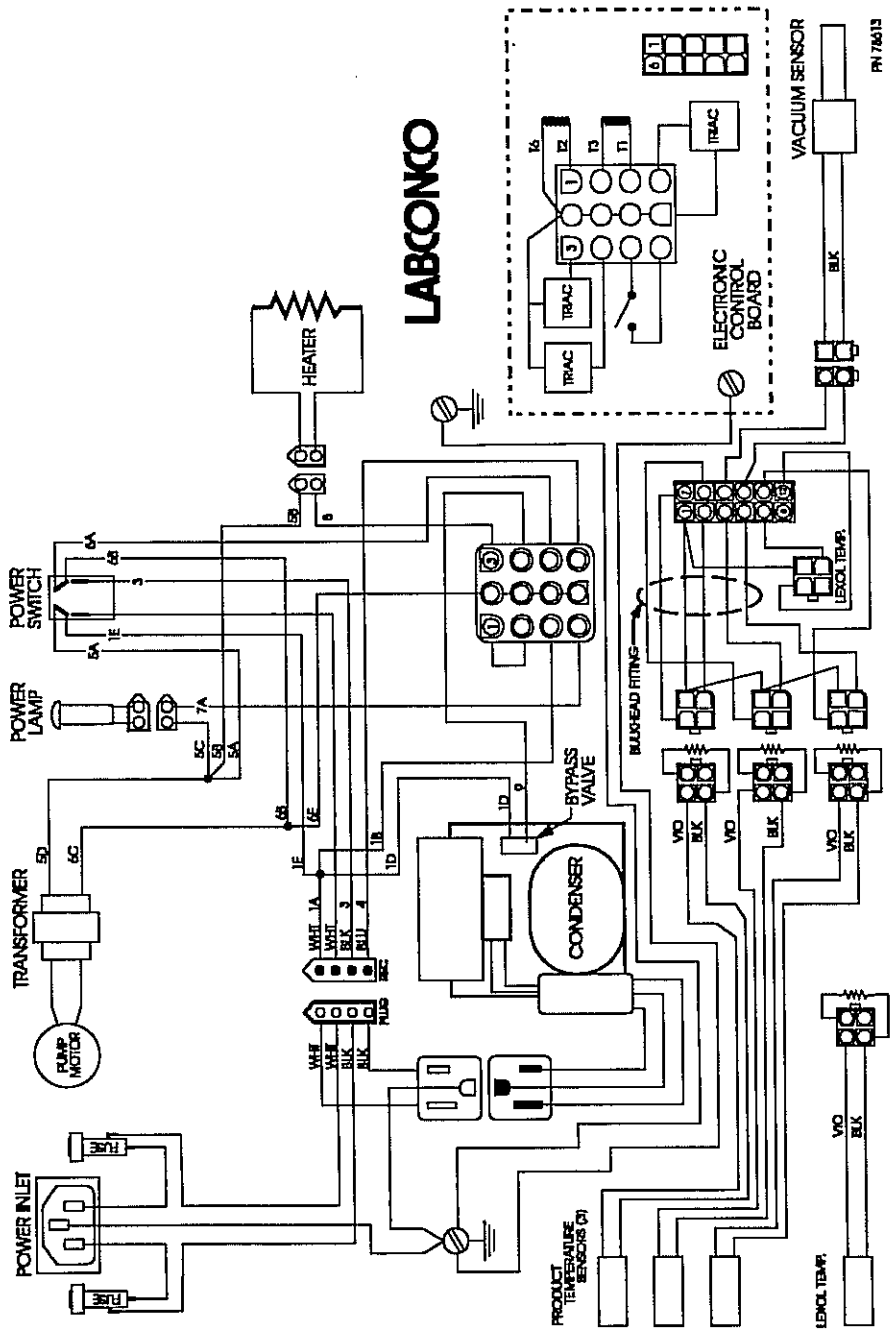


Figure 10

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 (If so equipped)	Close sample valve
	Purge valve closed (If so equipped)	Open purge valve
	Break or opening in vacuum lines or connections	Locate and repair
	Vacuum break control open	Move control to "close" position
Poor Vacuum (greater than 500 x 10^{-3}mBar)	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 or door gasket	Clean gasket and lid

PROBLEM	CAUSES	CORRECTIVE ACTION
Poor Vacuum (greater than 500 x 10^{-3}mBar) (Con't)	Damaged sample valve (If so equipped)	Locate and replace
Poor response of temperature control system	Fluid not circulating	Purge system of air or replace pump
	Refrigeration system solenoid valve not opening	Check connections to coil or replace coil, or replace valve
	Heater not operating	Check connections to heater, or replace heater
	Fluid level low	Fill fluid to line when unit is at ambient temperature and not under vacuum. Check for leaks.
Fluid level low	Lexol leaks	Inspect all tubes visually and by touch. Observe "kerosene" small in chamber. Observe oil in collector. Scan all connections and tubes with a black light.

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 circumstances 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.

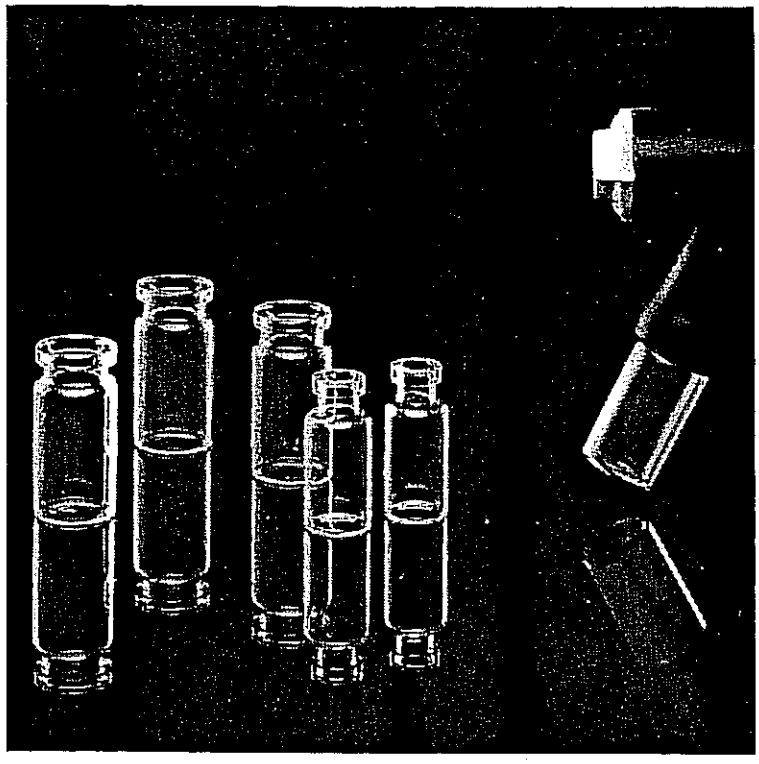
If a shipment is received in visibly damaged condition, be certain to make a notation on the delivering carrier's receipt and have his agent confirm the damage on your receipt. Otherwise, the damage claim may be refused.

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 NUMBER	DESCRIPTION
77561	**Tray With Slide-Out Bottom** 14" wide x 12" deep. Stainless steel tray has separate bottom which slides out allowing serum bottles and ampules direct contact with the shelf to facilitate the drying process.
77562	**Bulk Tray** 15" wide x 11-1/2" deep. Stainless steel tray will contain liquids forbulk drying.
77563	**Shelf Spacers** 2-1/2" high x 9" wide x 13" deep. Two stainless steel spacers placed on one or two empty shelves assist stoppering of partial loads of small vials or bottles.
75162	**Support Grid** 7" wide x 7" deep. Stainless steel tray with removable plastic grid provides support for Stoppering Ampules and other small specimen containers while stoppering under vacuum. Grid holds 144 ampules of 12 mm diameter.
77716	**Support Stand With Valves** 7-3/4" high x 30" wide x 15" deep. Supports Tray Dryer on any Lyph-Lock 18, 12 or 6 Liter Freeze Dry System. Six 3/4" valves on the stand provide the flexibility to connect flasks and other freeze dry glassware to the system. Can not be used with isolation valve 78600.
78600	**Isolation Valve** Stainless steel manually operated valve allows the vacuum in the Stoppering Tray Dryer to be isolated from the vacuum in the remainder of the Lyph-Lock Freeze Dry System. Can not be used with Support Stand with valves 77716.

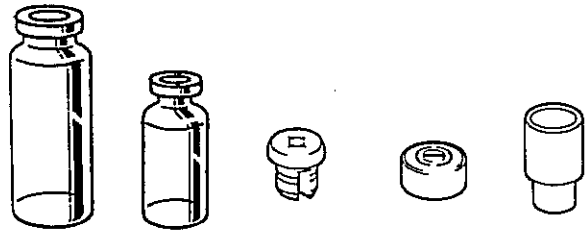
Serum Bottles and Vials



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

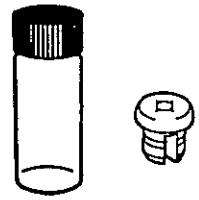
Seal Crimper

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.

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 Customer Service Department at either (800) 821-5525 or (816) 333-8811, between the hours of 8:00 a.m. and 5:00 p.m. Central Standard Time. For after hours emergency assistance, page Labconco from your touchtone phone. Call 1-800-821-5073, then 292-9508 followed by your area code and number. A Labconco Product Service Representative will return your call.