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User's Manual

FreeZone® Benchtop Shell Freezers

Models
79490 Series

To receive important product updates,
complete your product registration card
online at **register.labconco.com**

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

Warranty

Labconco Corporation provides a warranty to the original buyer for the repair or replacement of parts and reasonable labor as a result of normal and proper use of the equipment with compatible chemicals. Broken glassware and maintenance items, such as filters, gaskets, light bulbs, finishes and lubrication are not warranted. Excluded from warranty are products with improper installation, erratic electrical or utility supply, unauthorized repair and products used with incompatible chemicals.

The warranty for FreeZone® Benchtop Shell Freezers will expire one year from date of installation or two years from date of shipment from Labconco, whichever is sooner. Warranty is non-transferable and only applies to the owner (organization) of record.

Buyer is exclusively responsible for the set-up, installation, verification, decontamination or calibration of equipment. This limited warranty covers parts and labor, but not transportation and insurance charges. If the failure is determined to be covered under this warranty, the dealer or Labconco Corporation will authorize repair or replacement of all defective parts to restore the unit to operation. Repairs may be completed by 3rd party service agents approved by Labconco Corporation. Labconco Corporation reserves the rights to limit this warranty based on a service agent's travel, working hours, the site's entry restrictions and unobstructed access to serviceable components of the product.

Under no circumstances shall Labconco Corporation be liable for indirect, consequential, or special damages of any kind. This warranty is exclusive and in lieu of all other warranties whether oral, or implied.

Returned or Damaged Goods

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

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

Limitation of Liability

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

Contacting Labconco Corporation

If you have questions that are not addressed in this manual, or if you need technical assistance, contact Labconco's Customer Service Department or Labconco's Product Service Department at 1-800-821-5525 or 1-816-333-8811, between the hours of 7:30 a.m. and 5:30 p.m., Central Standard Time.

Part #7392209, Rev. C
ECO L427

TABLE OF CONTENTS

CHAPTER 1
INTRODUCTION

Congratulations on your purchase of a Labconco FreeZone® Benchtop Shell Freezer, which is designed to prepare samples for laboratory lyophilization procedures. The unit is easy to install and maintain. Proper care and maintenance of this product will result in many years of dependable service.

Freeze Dry Process

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

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

Freeze Dry Rates

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

Table 1

SAFE TEMPERATURE AND DRYING TIMES FOR SELECTED MATERIALS			
Material 10mm Thick	Safe Temperature °C	Collector Temperature °C	Hours (Approx.)
Milk	-5	-40	10
Urea	-7	-40	10
Blood Plasma	-10 to -25	-40	16
Serum	-25	-40	18
Vaccinia	-30 to -40	-50	22
Influenza Vaccine	-30	-50	24
Human Tissue	-30 to -40	-50	48
Vegetable Tissue	-50	-80	60

*Total sample quantities are contingent on various freeze dryer capacities.

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

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

In order for lyophilization to occur, ice must be removed from the frozen sample via sublimation. This is accomplished by the collector and the vacuum pump. The collector, which should be at least 15 to 20°C colder than the eutectic temperature (melting temperature) of the sample, traps vapor as ice. Since the vapor pressure at the collector is lower than that of the sample, the flow of water vapor is from the sample to the collector. Since this vapor diffusion process occurs very slowly under normal atmospheric conditions, a good vacuum is essential to maintain an efficient rate. In many applications, the maintenance of a vacuum of 0.133 mBar or less is recommended.

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

Freeze Dry Capacity

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

If there is a problem with a particular type of sample melting when placed on the freeze dry system, dilution of the sample with more water or providing some insulation around the flask to decrease the rate of heat absorption by the sample may help. If the eutectic temperature of the sample is –40 to –60°C, the freeze dry system selected for use must be equipped with cascade type refrigeration so that the collector temperature can be cooled to below –75°C, or a dry ice/solvent trap may be used between the collector and the vacuum pump.

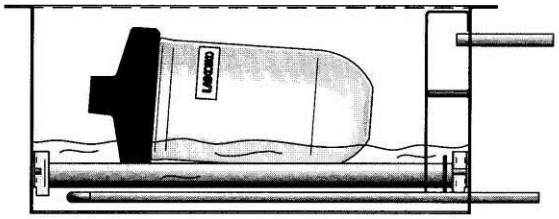
Samples Containing Volatile Substances

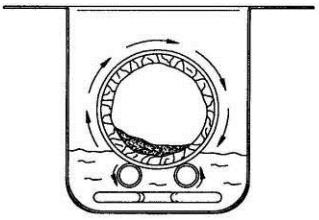
In certain cases the solvent in a sample to be freeze dried may contain volatile components such as acetonitrile, methanol, acetic acid, formic acid or pyridine. In addition to these substances having an effect on the eutectic temperature, they may increase the vapor pressure at the surface of the sample.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

Sample Surface Area and Thickness of the Sample

The volume and configuration of the suspension to be freeze dried often determines how the material will be freeze dried. For example, the greater the ratio of the surface area to the volume of the suspension, the faster drying will occur. This is because a greater area for the water molecules to leave the product exists compared to the distance they have to travel to reach the surface of the frozen matrix. Drying occurs from the top of the product and initially the removal of water molecules is efficient. However, as the drying front moves down through the product, drying becomes more and more difficult. The water molecules must now travel through the dried portions of the product, which impedes their progress. As the drying front moves further and further down the matrix, the application of heat to the product becomes more important.

Shell freezing as a method of prefreezing the product can increase the surface area-to-volume ratio by spreading out the frozen product inside the vessel. Shell freezing is accomplished by rotating the vessel in a low temperature bath causing the product to freeze in a thin layer on the inside surface of the vessel. The thickness of the frozen suspension depends on the volume of the product in comparison to the size of the vessel. The diagram below shows how a Shell Freezer operates.





About This Manual

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

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

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

Chapter 3: Getting Started contains the information you need to properly unpack, inspect and install your Benchtop Shell Freezer.

Chapter 4: Using Your Benchtop Shell Freezer discusses the basic operation and information on how to load samples.

Chapter 5: Maintaining Your Benchtop Shell Freezer explains how to perform routine maintenance.

Chapter 6: Troubleshooting contains information about problems you may encounter while using your Benchtop Shell Freezer, including the probable causes of the problems, and suggested corrective actions.

Appendix A: Benchtop Shell Freezer Components contains labeled diagrams of the key components of the Benchtop Shell Freezer.

Appendix B: Benchtop Shell Freezer Dimensions contains diagrams showing the dimensions for the Benchtop Shell Freezer.

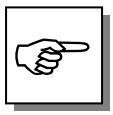
Appendix C: Benchtop Shell Freezer Specifications contains product specifications.

Typographical Conventions

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

- Book, chapter, and section titles are shown in italic type (e.g., *Chapter 3: Getting Started*).
- Steps required to perform a task are presented in a numbered format.
- Comments located in the margins provide suggestions, reminders, and references.

- Critical information is presented in boldface type in paragraphs that are preceded by the exclamation icon. Failure to comply with the information following an exclamation icon may result in injury to the user or permanent damage to your Shell Freezer.



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

CHAPTER 2
PREREQUISITES

Before you install your Benchtop Shell Freezer, you need to prepare your site for installation. The Benchtop Shell Freezer may be mounted on top of a benchtop work surface. Carefully examine the location where you intend to install your Benchtop Shell Freezer. You must be certain that the area is level and of solid construction. An electrical source must be located near the installation site. Carefully read this chapter to learn:

- the electrical supply requirements.
- location requirements.

Refer to *Appendix C: Benchtop Shell Freezer Specifications* for complete Benchtop Shell Freezer electrical and environmental conditions, specifications and requirements.

Electrical Requirements

The Benchtop Shell Freezer requires a dedicated electrical outlet. This outlet requires a 15 Amp circuit breaker or fuse for models rated at 115V (60 Hz). An outlet equipped with a 8 Amp circuit breaker or fuse is required for models rated at 230V (50/60 Hz). The power cord on 115V models is equipped with a 15 Amp NEMA 5-15P plug. Various power cord configurations are provided with 230V models. If this does not match with the available receptacle, remove this plug and replace it with an approved plug of the suitable style.

Location Requirements

The Benchtop Shell Freezer should be located in an area that provides an unobstructed flow of air around the cabinet. This air cools the refrigeration system. A minimum of 3" must be allowed between the back and both sides of the Benchtop Shell Freezer and adjacent wall surfaces. Restriction of airflow during operation could adversely affect performance.

Refer to *Appendix B: Benchtop Shell Freezer Dimensions* for dimensional drawings of the Benchtop Shell Freezer.

CHAPTER 3
GETTING STARTED

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

- unpack and move your Benchtop Shell Freezer.
- set up your Benchtop Shell Freezer.
- connect the electrical supply source to your Benchtop Shell Freezer.
- safely use solvents with your Benchtop Shell Freezer.

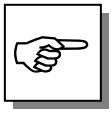
The Benchtop Shell Freezer weighs over 90 lbs. (41 Kg). The carton allows for lifting with a mechanical lift truck or hand truck. If you must lift the Benchtop Shell Freezer manually, use at least two (2) persons and follow safe lifting guidelines.

Unpacking Your Benchtop Shell Freezer

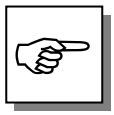
The United States Interstate Commerce Commission rules require that claims be filed with the delivery carrier within fifteen (15) days of delivery.

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

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



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



DO NOT DISCARD THE CARTON OR PACKING MATERIALS UNTIL YOU HAVE CHECKED ALL OF THE COMPONENTS AND INSTALLED AND TESTED THE BENCHTOP SHELL FREEZER.

Benchtop Shell Freezer Components

Locate the model of Benchtop Shell Freezer you received in the following table. Verify that the components listed are present and undamaged.

				Power Cords				
Catalog #	Description	Volts	Hz	1334500	1336100	1338000	1332600	1332700
7949020	Benchtop Shell Freezer	115	60	X				
7949030	Benchtop Shell Freezer	230	50		X			
7949040	Benchtop Shell Freezer	230	60			X		
7949060	Benchtop Shell Freezer	230	50				X	
7949070	Benchtop Shell Freezer	230	50					X

Plus the following:

Part #	Qty.	Component Description
7392209	1	User's Manual

If you did not receive one or more of the components listed for your Benchtop Shell Freezer, or if any of the components are damaged, contact Labconco Corporation immediately for further instructions.

Installing Your Benchtop Shell Freezer

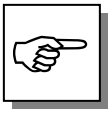
After you verify receipt of the proper components, move your Benchtop Shell Freezer to the location where you want to install it. Then, follow the steps listed below.

1. Check that the solvent bath drain plug is securely installed in the drain hose.
2. Remove bath compartment cover and add solvent to bath compartment. Commonly used solvents are methanol or ethanol. Recommended solvent depth is 1/4" to 1/2" above the top of the rollers (approximately 2 liters).

If flammable solvents are used in the shell freezer bath, be sure to keep away from open flame.

Electrical Connection

Plug the power cord into the receptacle on the back of the Benchtop Shell Freezer and plug the other end into a suitable power receptacle.



DO NOT ATTEMPT TO PLUG THE BENCHTOP SHELL FREEZER INTO A FREEZONE FREEZE DRY SYSTEM.

Solvent Safety Precautions

Solvents used in the Benchtop Shell Freezer may be flammable or hazardous to your health. Use extreme caution and keep sources of ignition away from the solvents.

Hazardous materials such as strong acids or bases, radioactive substances and volatile organics must be handled carefully and promptly cleaned up if spilled. If a sample is spilled, it must immediately be cleaned up.

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

CHAPTER 4
USING YOUR BENCHTOP SHELL FREEZER

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

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

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

Benchtop Shell Freezer Controls

The control panel for the Benchtop Shell Freezer is shown below with a description about its function.

1. Main Power Switch- (Not shown - Located on right side of cabinet) turns the unit on or off.
2. Bath Temperature Graph Display – This display indicates the temperature of the solvent in the Benchtop Shell Freezer bath.
   The top LED turns on when the main power switch is turned on.
   The second LED turns on when the bath temperature reaches approximately –20°C.
   The third LED turns on when the bath temperature reaches approximately –30°C.
   The fourth LED turns on at approximately –35°C.
   The fifth LED turns on at approximately –38°C.
   The sixth LED turns on at approximately –40°C.
   The bottom LED turns on at approximately –42°C.

Operating the Benchtop Shell Freezer

The Benchtop Shell Freezer is designed for shell freezing samples in flasks in preparation for freeze drying. It can process the following Labconco flasks

Quantity	Flask Size
1	1200 ml Flask
1	900 ml Flask
1	750 ml Flask
1	600 ml Flask
1	300 ml Flask
2	150 ml Flask
2	120 ml Flask
2	80 ml Flask
2	40 ml Flask

Follow the steps below each time you use the shell freezer to obtain optimum performance:

1. Check that the solvent bath drain plug is securely installed in the drain hose.
2. Remove the bath compartment cover and add solvent to the bath compartment. Commonly used solvents are methanol or ethanol. Recommended solvent depth is 1/4" to 1/2" above the top of the rollers (approximately 2 liters).

If flammable solvents are used in the shell freezer bath, be sure to keep away from open flame.

3. Press the main power switch. The top LED will illuminate. The shell freezer refrigeration module will start and the bath rollers will begin rotating.
4. Fill a freeze dry container no more than ½ full with sample and stopper the container top. Lay the container on the rollers in a horizontal position. Replace the bath compartment cover during shell freezing.
5. When the sample is completely frozen in the container, remove the container from the bath compartment and freeze dry the sample or place it in a suitable storage freezer for future freeze drying.
6. Press the main power switch to turn shell freezer off.
7. If the Benchtop Shell Freezer is to be used for cold bath operation, additional solvent may be added to submerge small flasks. Do not fill pan more than 2 inches below the top of the stainless steel pan with vessels submerged. Allow additional time for larger volumes of solvent to reach lower temperatures.

8. **Always drain flammable solvents from the bath when the shell freezer is not in use and store the solvent in a suitable container. This is accomplished by first pulling the drain hose out of the left side panel of the unit and then removing the drain plug. Place the hose in a suitable container to collect the solvent**.

CHAPTER 5
MAINTAINING YOUR BENCHTOP SHELL FREEZER

Under normal operation, the Benchtop Shell Freezer requires little maintenance. The following maintenance schedule is recommended:

As needed:

1. If the heat transfer liquid in the bath becomes contaminated, drain and replace. It is the user's responsibility to dispose of it in accordance with all applicable regulations.
2. Clean up all spills.
3. Clean the lid and gasket using a soft cloth, sponge or chamois and a mild, non-abrasive soap or detergent.

Monthly:

1. Check the drain hose and lid gasket and replace if they 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 bath lid.
3. Using a soft cloth, sponge, or chamois and a mild, non-abrasive soap or detergent, clean the exterior surfaces of the unit. Liquid spray cleaners and polishes may be used on the exterior surfaces. Do not use solvents to remove stains from the exterior surfaces as they may damage the finish.

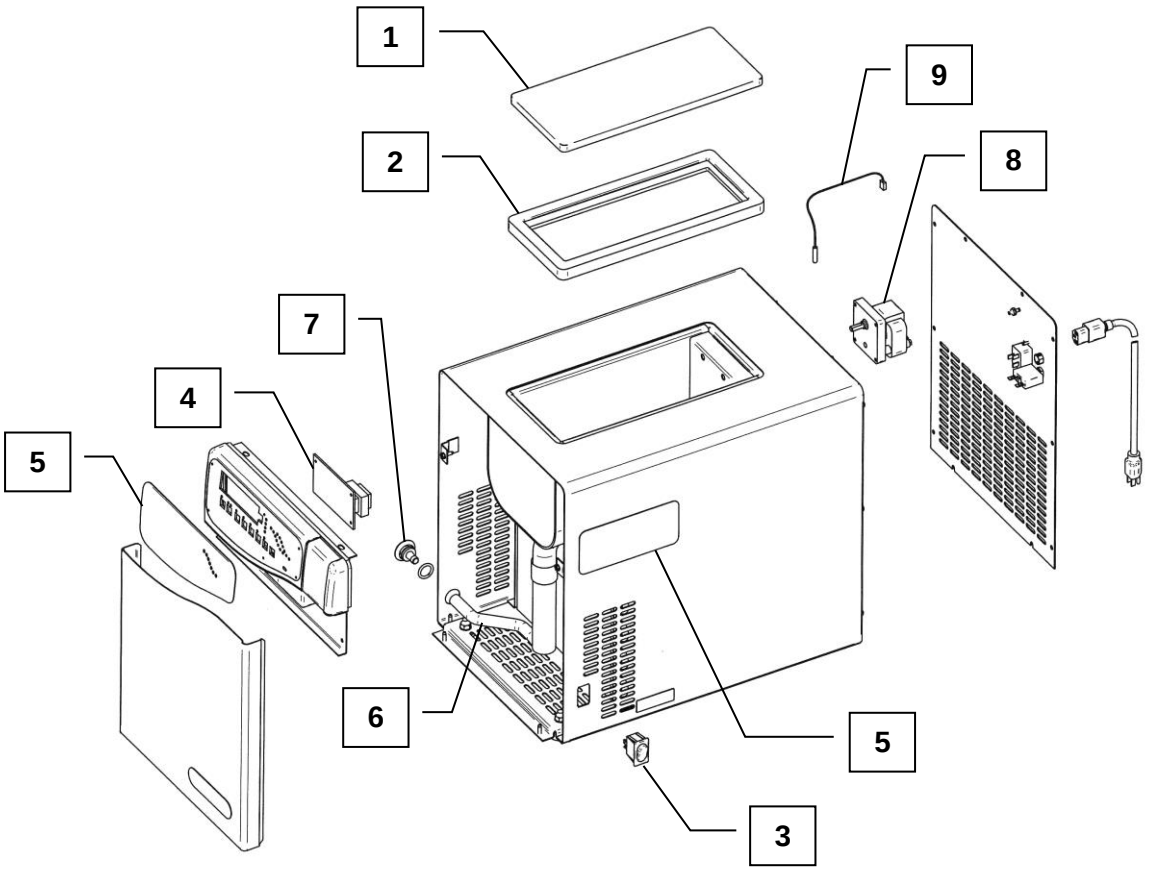
Annually:

1. Every 12 months, or more often if the Benchtop Shell Freezer is operated in a dusty environment, the refrigeration system condenser should be cleaned. Using a vacuum cleaner and brush attachment, clean the condenser to ensure proper airflow for peak performance. It can be accessed by removing the front lower panel.

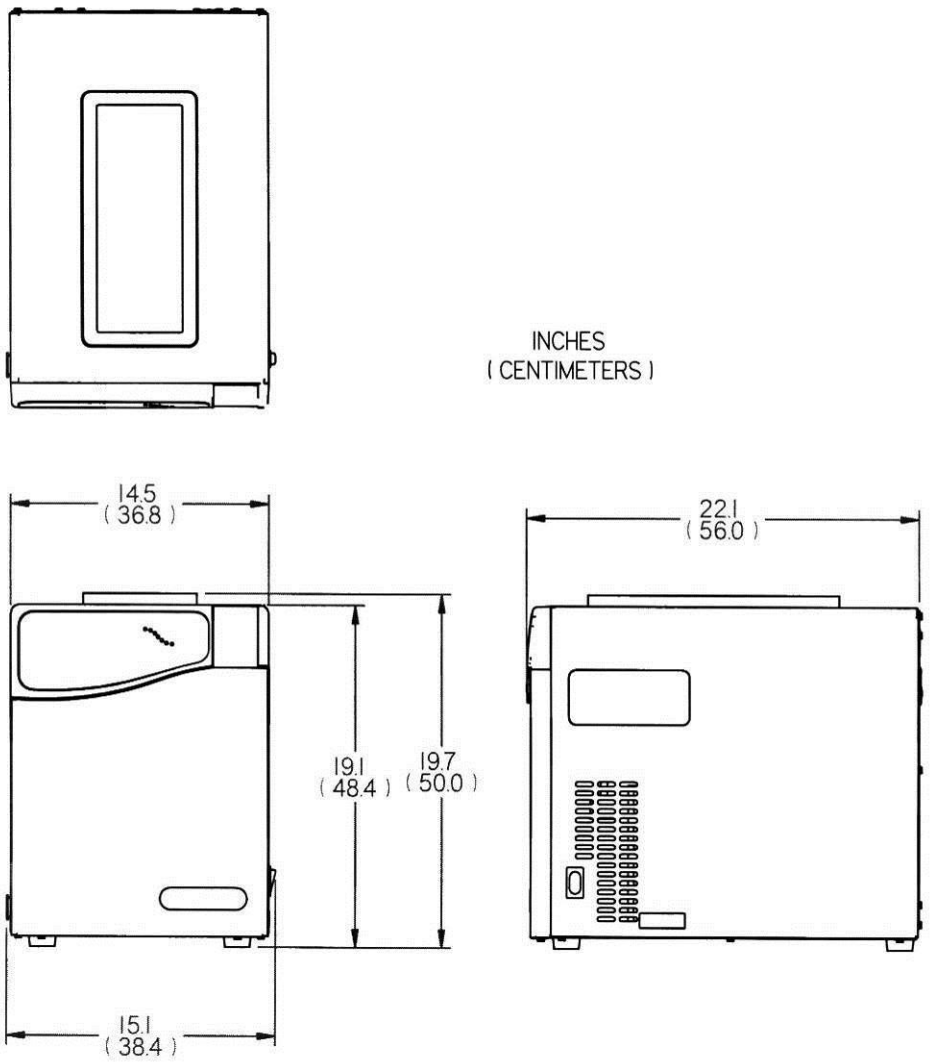
APPENDIX A
BENCHTOP SHELL FREEZER COMPONENTS

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

Item	Qty	Part No.	Description
1	1	7403100	Lid
2	1	7408400	Gasket – Lid
3	1	1305200	Switch
4	1	7394400	Printed Circuit Board
5	1	7392208	Label
6	1	7624100	Hose – Drain
7	1	7728000	Plug – Drain
8	1	7519100	Drive Motor 115V
		7519101	Drive Motor 230V
9	1	7515300	Temperature Sensor



APPENDIX B
BENCHTOP SHELL FREEZER DIMENSIONS



APPENDIX C
BENCHTOP SHELL FREEZER ELECTRICAL SPECIFICATIONS

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

Electrical Specifications

Catalog Number	Nominal	Operating Range	Frequency	Phase	Amperage
7949020	115	103-127	60	1	6
	100	90-110	50	1	6
7949030	230	198-254	50	1	2.5
7949040	230	187-253	60	1	2.5
7949060	230	187-253	60	1	2.5
7949070	230	187-253	60	1	2.5

Environmental Conditions

- Indoor use only.
- Maximum altitude: 6562 feet (2000 meters).
- Ambient temperature range: 41° to 104°F (5° to 40°C).
- Maximum relative humidity: 80% for temperatures up to 88°F (31°C), decreasing linearly to 50% relative humidity at 104°F (40°C).
- Main supply voltage fluctuations not to exceed ±10% of the nominal voltage.
- Transient over voltages according to Installation Categories II (Over voltage Categories per IEC 1010). Temporary voltage spikes on the AC input line that may be as high as 1500V for 115V models and 2500V for 230V models are allowed.
- Used in an environment of Pollution degrees 2 (i.e., where normally only non-conductive atmospheres are present).

  Occasionally, however, a temporary conductivity caused by condensation must be expected, in accordance with IEC 664.