



SP INDUSTRIES, INC.

Freeze Dryer Operator's Manual



VirTis BenchTop 2K, 4K and 6K Freeze Dryers

SP Service

VirTis

Hull

Wilma|LabGlass

Genevac

FTS

Hotpack

Copyright © 2009 SP Industries, Inc. All marks herein are used under license.
All brand or product names mentioned may be trademarks or registered trademarks of their respective companies.

Part Number 414258
Revision 004, 12/09

| | | | |
|---------------------------|-------------------------------|------------------------|---------------------------|
| SP Industries Inc. | Corporate Headquarters | | |
| | 935 Mearns Road | 815 State Route 208 | 3538 Main Street |
| | Warminster, PA 18974 USA | Gardiner, NY 12525 USA | Stone Ridge, NY 12484 USA |
| | (800) 523-2327 | (800) 431-8232 | (800) 824-0400 |
| | (215) 672-7800 | (800) 944-8559 | (845) 687-0071 |
| | | (845) 255-5000 | |

SP Service (877) 548-4666
SP Service Fax (215) 672-1702

Website www.spindustries.com

This **Freeze Dryer Operator's Manual** contains confidential and proprietary information of SP Industries, Inc. and may be used only by a recipient designated by and for purposes specified by SP Industries, Inc.

Reproduction of, dissemination of, modifications to, or the creation of derivative works from this **Freeze Dryer Operator's Manual**, by any means and in any form or manner, is expressly prohibited, except with the prior written permission of SP Industries, Inc. Permitted copies of this document must retain all proprietary notices contained in the original.

The information in this document is subject to change without prior notice. Always confirm with SP Industries, Inc. that you are using the most current version of this document. SP Industries, Inc. is free to modify any of its products and services, in any manner and at any time, notwithstanding the information contained in this document.

THE CONTENTS OF THIS DOCUMENT SHALL NOT CONSTITUTE ANY WARRANTY OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE IMPLIED WARRANTIES OF MERCHANTABILITY AND/OR FITNESS FOR A PARTICULAR PURPOSE OR GIVE RISE TO ANY LIABILITY OF SP INDUSTRIES, INC., ITS AFFILIATES OR ITS SUPPLIERS.

The terms and conditions governing the use of this **Freeze Dryer Operator's Manual** shall consist of those set forth in written agreements with SP Industries, Inc.



*Committed to Product Excellence
...and Continuous Support*

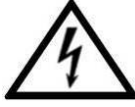
Important Symbols



Warning! Injury or even death may result if a recommendation marked with this symbol is not heeded.



Crush Hazard. Keep hands clear when operating door.



Electric Shock Danger! Use appropriate caution to avoid injury or death.



Corrosive Chemical. Wear suitable gloves, safety glasses, and protective clothing.



Burn Danger! Potentially hot surface. Use appropriate caution.



Property Caution! To prevent damage to chamber equipment and/or load, adhere to procedures marked by this symbol.



Do not store flammable materials in chamber.



Practical Operating Tip. These recommendations streamline unit operation and prevent common operator errors.



Wear Safety Glasses.



Explosive Materials Hazard! Keep objects away from heat.

Freeze Dryer Safety Warnings

- ✓ *Always assume that shelf, condenser and internal parts may be very cold or very hot. Wear protective equipment to avoid burns.*
- ✓ *Always ensure that only an authorized technician services the refrigeration, heat transfer, vacuum and electrical systems.*
- ✓ *Always ensure that refrigeration air intake is clear and clean.*
- ✓ *Always ensure vacuum pump exhaust is properly ventilated and/or contained.*
- ✓ *Always practice team lifting when moving heavy equipment.*
- ✓ *Always use a maximum one pound regulator if backfilling from an inert gas source.*
- ✓ *Always wear safety glasses when using glass flasks.*
- ✓ *Do carefully read the entire instruction manual before attempting to operate the freeze dryer.*
- ✓ *Do verify that the electric service and other utilities match the unit's requirements before connecting to power.*
- ✓ *Never allow hand or body contact with open vacuum ports.*
- ✓ *Never clean with solvents. Use mild detergent and water only.*
- ✓ *Never operate the unit without all covers in place.*
- ✓ *Never pressurize chambers. Laboratory freeze-drying systems are designed for vacuum only.*
- ✓ *Never stopper vials unless the chamber door is tightly closed.*
- ✓ *Never use acrylic closures if they are cracked or crazed.*
- ✓ *Never use with toxic, corrosive, flammable or organic materials unless special precautions are in place to prevent injury to personnel or damage to equipment.*

Warranty Information

VirTis BenchTop 2K, 4K and 6K are warranted by SP Industries to be free of defects in material and workmanship when operated under normal conditions as specified in the instructions provided in this manual. Please take this opportunity to locate the serial tag on your new VirTis BenchTop 2K, 4K and 6K and record the information below for future reference. SP Industries also recommends that you complete and return your unit's warranty registration card.

Model Number _____

Serial Number _____

Part Number _____

Limited Warranty

SP Industries (the "Company") warrants each of its products against any defects in material or workmanship, provided that the product is used in a reasonable manner under appropriate conditions and consistent with the applicable operating instructions, for a period of 12 months from the date of installation or 15 months from the date of shipment (whichever comes first).

The obligation of the Company shall be, at its option, to repair or replace, without charge any parts that prove to be defective within the warranty period, if the purchaser notifies the Company promptly in writing of such defect. The Company shall not be responsible for labor charges payable with respect to persons other than Company employees. Replacement or repair of parts pursuant to this warranty shall not in any way extend the original warranty period. The Company will not be responsible for any unauthorized repairs, replacements or product modifications, nor will it be responsible for any product failures resulting from such unauthorized repairs, replacements or product modifications negligently or otherwise made by persons other than Company employees or authorized representatives of the Company.

THE COMPANY DOES NOT MAKE AND EXPRESSLY DISCLAIMS ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE OR ANY OTHER WARRANTY, EXPRESSED OR IMPLIED, WITH RESPECT TO THE SALE, INSTALLATION, DESIGN OR USE OF ITS PRODUCTS. ADDITIONALLY, THE COMPANY SHALL NOT BE LIABLE FOR ANY CONSEQUENTIAL DAMAGES RESULTING FROM THE USE OF OR ANY DEFECTS IN ITS PRODUCTS.

The Company's employees are available to provide general advice to customers concerning the use of the Company's products; however, oral representations are not warranties with respect to particular products or their uses and may not be relied upon if they are inconsistent with the relevant product specifications for the items set forth herein.

Notwithstanding the above, the terms and conditions set forth in the Company's formal sales contracts shall be controlling and supersede any inconsistent terms contained herein, and any changes to such contracts must be made in writing and signed by an authorized executive of the Company.

Contents

| | |
|--|-----------|
| Important Symbols | ii |
| Freeze Dryer Safety Warnings | ii |
| Warranty Information | iii |
| Introduction..... | 1 |
| Key Features..... | 1 |
| BenchTop Usage | 1 |
| Available Configurations | 2 |
| From Freeze Dryer to Condenser Module..... | 2 |
| Cold Trap Condenser Module | 2 |
| Installation and Setup | 3 |
| Initial Inspection | 3 |
| Service Connections | 3 |
| Vacuum Pump Installation | 4 |
| Installing the Vacuum Pump..... | 4 |
| Oil Mist Eliminators..... | 5 |
| Freeze Dryer Setup..... | 6 |
| Vacuum Baffle Plate | 7 |
| Freeze-Drying Concepts | 9 |
| Elements of Freeze-Drying | 9 |
| Freeze | 9 |
| Vacuum | 9 |
| Heat..... | 10 |
| Condenser..... | 10 |
| Product Dependencies..... | 10 |
| Sentry 2.0 Controls..... | 11 |
| LED Wave Display (Qualitative Reference) | 11 |
| Button Functions | 12 |
| Up/Down (+/-) Buttons..... | 12 |
| MENU Button | 12 |
| COND Button | 12 |
| VAC Button..... | 12 |
| AUTO Button | 12 |
| DEFR Button | 12 |
| LCD Display Functions | 13 |
| Main Menu | 13 |
| Vacuum Control..... | 14 |
| Purge (Optional) | 14 |
| Heat (Optional) | 14 |
| User Options Menu | 14 |
| Vacuum Control..... | 14 |
| Temperature Units..... | 15 |
| Vacuum Units | 15 |

| | |
|---|-----------|
| Relay Test | 15 |
| Vacuum Pump Maintenance | 15 |
| Alarms Menu | 16 |
| Setpoints Menu | 16 |
| Condenser Temperature | 16 |
| Vacuum OK | 16 |
| Power Save Deadband | 16 |
| Maintenance Menu and Factory Options | 17 |
| Factory Options | 17 |
| Interstage Control | 18 |
| Calibration | 18 |
| Basic Operation | 21 |
| Getting Started | 21 |
| Product Preparation: Flask / Manifold Drying | 21 |
| Product Preparation: Drum Manifold Shelf Drying | 21 |
| Product Preparation: Chamber Drying | 22 |
| Quickseal Valves | 22 |
| Operation Instructions | 23 |
| Meltback | 24 |
| Preventing Glassware Breakage | 25 |
| Product Dryness End Point | 25 |
| Stoppering (Optional) | 25 |
| Cold Trap Condenser | 27 |
| Product Requirements | 27 |
| Cold Trap Setup | 28 |
| Optional Components | 29 |
| Manifolds | 29 |
| Shelf Racks | 30 |
| Liquid Nitrogen Trap | 31 |
| Filter Trap | 32 |
| BenchTop Cold Plate | 33 |
| Initial Setup of the BenchTop Cold Plate | 33 |
| Cold Plate Operation | 33 |
| General Maintenance | 35 |
| Vacuum System | 35 |
| Vacuum Pump | 35 |
| Changing Vacuum Pump Oil | 35 |
| Vacuum Tubing and Gaskets | 36 |
| Door Gasket | 36 |
| Vacuum Pump Gas Ballast Valve | 36 |
| Quickseal Valves | 37 |
| Refrigeration System | 37 |
| The Air-Cooled Condenser | 37 |
| Condenser Chamber | 38 |
| Acrylic Parts | 38 |
| Appendix A: Troubleshooting | 39 |
| Vacuum Problems | 39 |
| Product Melting | 40 |
| Appendix B: Serum Vial Capacities | 41 |
| Appendix C: Stainless Steel Cleaners | 43 |

Appendix D: Spare Parts List..... 45

Introduction

VirTis BenchTop 2K, 4K and 6K freeze dryers offer a low-cost solution for laboratories with space constraints. Featuring condenser temperatures of -55 °C to -105 °C, BenchTop freeze dryers can handle a wide range of processing requirements.

Key Features

- Extremely compact design.
- Direct chamber, flask and/or shelf drying capabilities.
- Versatile Sentry 2.0 control panel.
- Three- to nine-liter condenser capacity.
- Variety of refrigeration system options.
- Digital display of temperatures and setpoints.
- Quickseal valves for processing flasks.¹
- Manual shelf stoppering (optional).

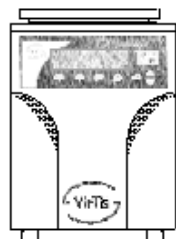
BenchTop Usage

BenchTop units are mechanically refrigerated condenser modules that can be utilized as freeze dryers or cold traps. These versatile units can also be used for initial product freezing.

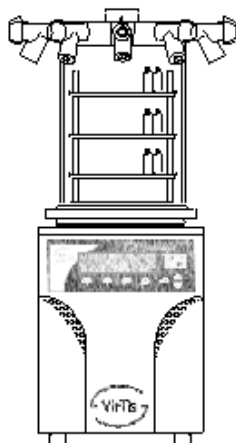
When used in freeze-drying mode, the BenchTop unit can effectively remove up to 99% of product moisture. The cold trap condenser module is capable of trapping vapors off of a vacuum concentrator or gel dryer.

¹ Requires optional manifold.

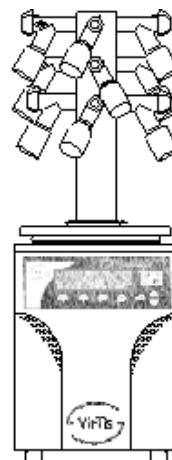
Available Configurations



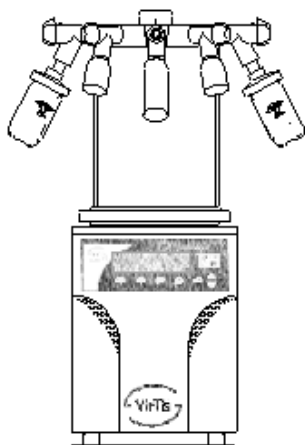
Freeze-drying in the condenser



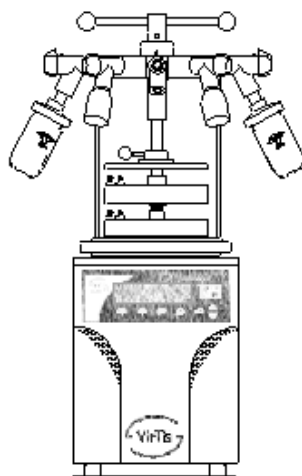
Shelf unit in drum manifold



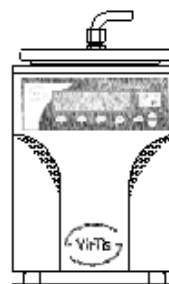
Vertical manifold



Flask drying on drum manifold



Flask drying on drum manifold with stoppered shelves



Fitted for cold trap application

From Freeze Dryer to Condenser Module

When using your BenchTop as a condenser module, the vacuum cover plate is replaced with either a $\frac{3}{4}$ -inch stainless steel port for the manifold plate or a plain cover plate, which must be purchased separately if no manifold is ordered. The manifold adapter plate is required when any manifold is purchased.

Cold Trap Condenser Module

When using a vacuum concentrator or gel dryer with your BenchTop unit, the condenser module can be used as a cold trap, which traps vapors driven off of the product before they reach the vacuum pump. For more information, refer to Chapter 6: Cold Trap Condenser.

Note: A low temperature condenser is required when trying to trap organic solvents.

Installation and Setup

Initial Inspection

Upon receiving your BenchTop series freeze dryer, inspect all contents of your shipment for damage. Check packing material for small accessory items. Remove all packing material carefully and inspect the unit for shipping damage. In the event that shipping damage has occurred, retain all packing material and contact your freight carrier immediately.

Note: *In the event of shipping damage, SP Industries will cooperate in the matter of collecting your claim, but is not responsible for the collection or free replacement of material. When possible, replacement parts will be shipped and invoiced to you, making them a part of your claim.*

Service Connections

Make sure that the outlet you intend to use meets the voltage and amperage requirements listed on the serial tag of your unit.



CAUTION! ONLY A QUALIFIED ELECTRICIAN SHOULD CONNECT THE UNIT TO THE AVAILABLE ELECTRICAL SUPPLY.

The line cord has three individual conductors inside the outer jacket.² To make the appropriate plug connection:

1. Trim back enough of the jacket to facilitate installation of the plug.
2. The three individual conductors are BROWN, BLUE, and GREEN with a YELLOW tracer.
3. Connect the BROWN wire to the line (hot) terminal on the plug.
4. Connect the BLUE wire to the neutral terminal.
5. Connect the GREEN/YELLOW wire to the ground terminal.

² Consult SP Industries and a qualified electrician if electrical configurations vary from standard, specified service requirements.

Vacuum Pump Installation

A remotely mounted vacuum pump is required for operation of your BenchTop freeze dryer. The vacuum pump must be a two-stage, high-vacuum pump that does not exceed the maximum allowable amperage listed on the unit's serial tag and back panel label.

The following parts are included with your BenchTop unit for connection to the vacuum pump:

- Four (4) feet of 3/4-inch ID (Inside Diameter) rubber vacuum tubing
- One (1) 90° rubber elbow
- One (1) plastic connector

Installing the Vacuum Pump

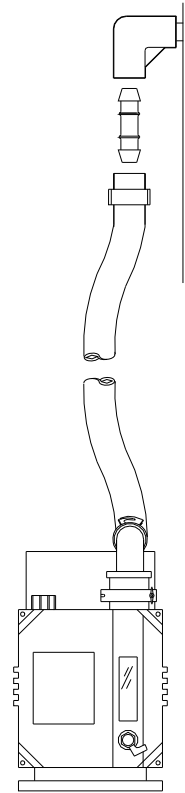
Notes: If you are installing a previously used vacuum pump, refer to the vacuum pump manual and the General Maintenance section of this manual. Ensure that the pump was properly maintained prior to installation.

A vacuum pump inlet port adapter and sufficient tubing are required for connection to the vacuum pump. If you need assistance, please contact SP Industries.

1. Position the vacuum pump in a convenient location near the freeze dryer. Ensure that the pump will be easily accessible during routine maintenance.
2. Cut the supplied vacuum tubing as short as possible. Allow enough length between the vacuum pump and the BenchTop unit.

Note: Clamp and cut the 3/4-inch ID vacuum hose. If you do not wish to cut the hose, it may be used at its full length, but may take up more space than necessary.

3. Locate the inlet port on your vacuum pump. Refer to the vacuum pump manual.
4. Remove any material with the exception of the inlet filter screen and gasket.
5. Place the adapter on the inlet port and secure with a fitting. If an adapter is not present, contact SP Industries.
6. Remove all objects from the vacuum pump outlet port, but retain for future use.
7. Connect the BenchTop unit to the vacuum pump using the 3/4-inch vacuum tubing. If connecting to a vacuum pump purchased from SP Industries, attach the 3/4-inch tubing from the BenchTop to the vacuum pump intake nipple, add a tubing clamp and tighten securely.
8. Check the vacuum pump oil level. The oil level should read half way up the sight glass. Add oil only if necessary. **DO NOT OVERFILL!**
9. Verify that the power switch on the vacuum is off. The LED next to the **VAC** button on the unit's front panel must be off.



Vacuum pump

10. Plug the vacuum pump into the receptacle marked VACUUM PUMP on the back of the unit. The receptacle is an IEC universal outlet. This allows you to control the pump using the Sentry 2.0. The voltage, phase and frequency of the vacuum pump must match the voltage, phase and frequency specified on the rear panel of the freeze dryer.

Note: You can power your vacuum pump from a wall socket if it does not have an IEC connector, but you will not be able to control the pump from the Sentry 2.0 Controller. Only a qualified electrician should perform installation of an IEC connector. The pump used should not exceed the amperage listed on the BenchTop serial tag.

11. Enable power to the vacuum pump by switching the pump's circuit breaker to the on position.

Oil Mist Eliminators

To reduce fumes from the vacuum pump and/or vent the vacuum exhaust externally, SP Industries recommends the installation an Oil Mist Eliminator (OME).

Related Parts

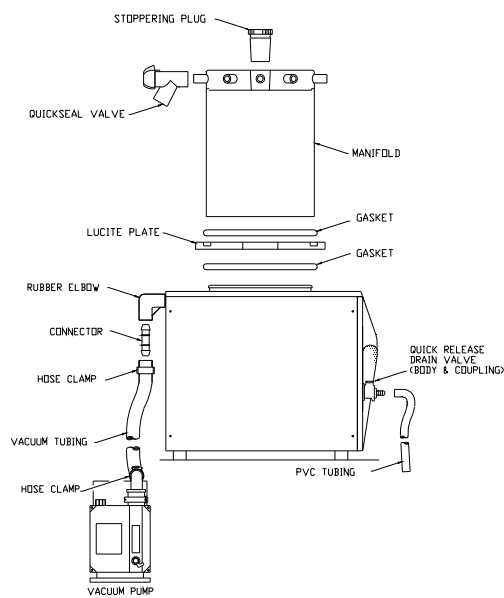
- Oil Mist Eliminator, Plastic for Oerlikon Leybold TRIVAC E D2.5E Vacuum Pump (P/N 414203).

Freeze Dryer Setup

To set up your BenchTop unit:

Note: Refer to *Basic Operation* for complete equipment operation instructions.

1. Install a condenser gasket (black rubber ring with a slit in it) on the top of the unit. It should have a light film of vacuum grease on the outer surface.
2. Prepare the unit for your intended use.
 - a. If the unit is to be used as a standalone freezer, cover the condenser with a plain cover plate to prevent air circulation from warming the samples. Skip to step 5.
 - b. If the unit is to be used as a manifold dryer, place a manifold adapter plate (clear acrylic or stainless steel circular disk with a hole) over the gasket.
 - c. If you are using an acrylic drum manifold, place a second condenser gasket around the bottom lip of the manifold.
 - d. If you are using a stainless steel manifold, place the supplied O-ring in the groove of the manifold adapter plate.



BenchTop with acrylic manifold.

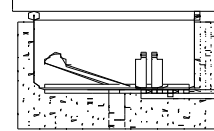
3. If using a manifold, attach Quickseal valves to each port on the manifold and ensure that each is in the closed position. For more information, refer to the Quickseal valve section in Chapter 5.

Note: Quickseal valves are siliconized at the factory. Vacuum grease is not required.
4. If using an acrylic drum manifold, apply a thin coating of vacuum grease to the tapered surface and place the tapered plug in the top center of the manifold. Slowly turn the plug 360° clockwise and then 360° counterclockwise. This will ensure a good seal between the surface of the plug and the manifold.
5. Plug the unit into an appropriate outlet and switch on the circuit breaker located on the rear of the unit. The control panel display will illuminate.

Vacuum Baffle Plate

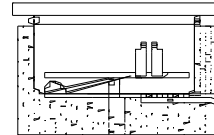
The BenchTop 2K and 4K models include a two-position vacuum baffle plate.

- **Baffle Plate on Chamber Bottom (Position 1).**
By raising the baffle plate handle, the plate can be dropped to the bottom of the chamber. This allows you to pre-freeze items on the baffle plate. In some cases, it may be more convenient to remove the baffle plate completely and freeze items directly on the bottom of the chamber.



Note: Freeze-drying cannot be performed with the baffle on the chamber bottom (position 1) since the vacuum port will be blocked and the product will remain too cold for water vapor transfer.

- **Baffle Plate in Raised Position (Position 2).**
Lower the baffle plate handle to raise the baffle plate when freeze-drying products directly on the baffle. This allows ice to build up on the condenser below and provides more room for the materials to be dried.



Freeze-Drying Concepts

The first step of lyophilization (freeze-drying) is typically to freeze the product to a minimum of $-40\text{ }^{\circ}\text{C}$. After the product is frozen, moisture can be extracted through sublimation. The sublimation process takes a solid (*i.e.*, ice) and converts it to a gas (*i.e.*, vapor), bypassing the liquid stage. The moisture is converted to a gas, while maintaining the material's crystalline structure.

The second step involves the migration of moisture from the product. BenchTop series freeze dryers facilitate the migration of moisture by using a vacuum to extract non-condensable vapors in the product chamber or container. The freeze dryer creates a low-pressure zone to allow moisture to flow easily from the product. Since vapor naturally travels toward cooler surfaces, the condenser chamber is cooled to at least $15\text{ }^{\circ}\text{C}$ below that of the product to attract product moisture.

Many products require a secondary drying process to remove moisture that did not convert to vapor during the first process. During secondary drying, BenchTop units remove the remaining bound moisture by slightly warming the product.

Note: To prevent meltback, the product should not be warmed above its eutectic point until all free ice has sublimated.

Elements of Freeze-Drying

Freeze

The product must be completely frozen before freeze-drying can begin. Product freezing can be accomplished directly in the condenser chamber, or through pre-freezing methods such as dry-ice baths or a laboratory freezer. The product's frozen temperature should be at least $-40\text{ }^{\circ}\text{C}$. To minimize freeze-drying time and protect the product against meltback, the product should be frozen in the thinnest cross section possible.

Vacuum

Adding vacuum assists in removing air and other non-condensable vapors from the chamber to facilitate vapor migration. The vacuum system creates a nearly pressure-free environment to allow the vapor from the frozen product to flow toward the cold condenser surface easily.

Heat

Carefully controlled heat input to the frozen product speeds the drying process. The BenchTop unit is designed to be used as either a manifold freeze dryer or a tray dryer. Exposing glassware containing frozen product to ambient room temperature helps drive the drying process by adding heat. This causes the frozen solvents in the product to sublime and then migrate to the colder surface of the condenser wall.

Several heated and unheated racks and stoppering accessories are available for freeze-drying in drum manifolds.

In its simplest configuration, a BenchTop unit will freeze-dry products on the partially raised vacuum baffle in the center of the condenser. Heat will be added by radiation through the acrylic cover.

Condenser

Trapping water vapor molecules in the form of ice on the condenser surface effectively removes moisture from the product. The refrigeration system cools the internal condenser at the bottom of the chamber to attract and trap vapors migrating off the product.

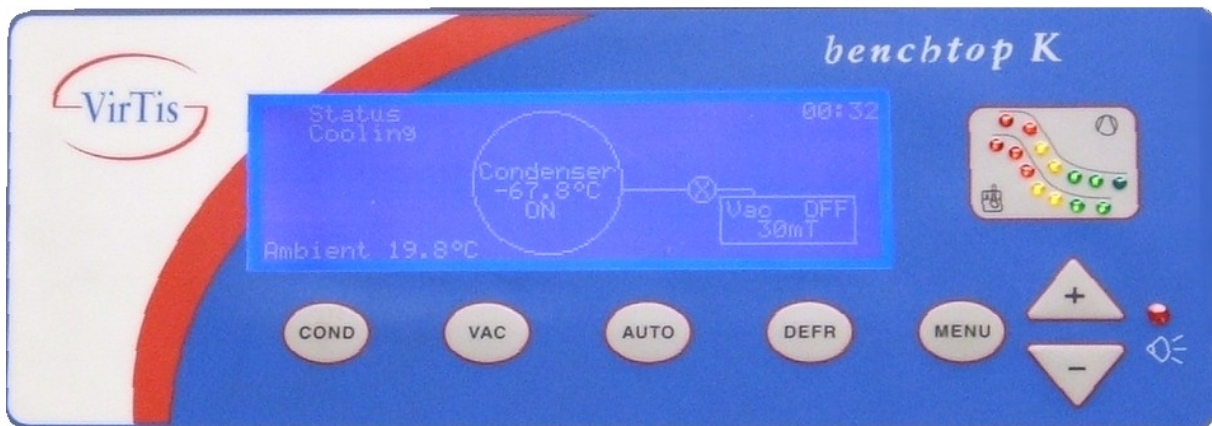
The condenser is located under the manifold to provide the shortest vapor path to achieve the maximum drying rate. The condenser capacity ranges from 3 to 9 liters, which indicates the maximum volume that can be condensed from the product before defrosting is necessary.

Product Dependencies

The freeze-drying process is completely dependent upon (and will change with) each specific product. In all cases, the condenser temperature must be cold enough to trap the vapors migrating from the product effectively. If the condenser is not cold enough, excess vapors may be pulled into the vacuum pump inadvertently. To compensate for this issue, SP Industries offers optional filter and liquid nitrogen ($N_{2(liq)}$) traps.

Sentry 2.0 Controls

The Sentry 2.0 is the control center for the BenchTop K series. Driven by a powerful microprocessor, the Sentry 2.0 is extremely versatile while remaining intuitively easy to use. The controller allows custom programming, includes a detailed LCD display and offers a wealth of customizable features.



The Sentry 2.0 front panel consists of the following:

- An LED wave display, which allows you to assess the status of the system quickly and easily.
- Tactile buttons located on the membrane, which allow you to activate specific functions and navigate through the menus within the LCD display.
- An LCD display, which shows the status of the unit and allows you to adjust the alarm and control parameters, as well as modify system configurations.

LED Wave Display (Qualitative Reference)

1. Condenser Temperature

The LEDs on the bottom wave represent the following temperatures:

| LED | Temperature (°C) | Color |
|-----|------------------|-------|
| 1 | 10 | Red |
| 2 | 0 | Red |
| 3 | -10 | Red |
| 4 | -20 | Amber |
| 5 | -30 | Amber |
| 6 | -40 | Green |
| 7 | ≤ -50 | Green |

2. Vacuum

The LEDs on the top wave represent the following vacuum setpoints:

| LED | Vacuum (mTorr) | Color |
|-----|----------------|-------|
| 1 | 2000 | Red |
| 2 | 1000 | Red |
| 3 | 500 | Amber |
| 4 | 200 | Amber |
| 5 | 100 | Green |
| 6 | 50 | Green |
| 7 | ≤ 20 | Green |

Button Functions

Up/Down (+/-) Buttons

The **Up/Down (+/-)** buttons allow you to navigate the menus and increase or decrease setpoint values displayed on the LCD display.

MENU Button

The **MENU** button performs a number of functions. During an alarm condition, pressing the **MENU** button will temporarily silence (but not clear) the alarm. When viewing the Synoptic screen, the **MENU** button will display the Main Menu.

The **MENU** button also acts as an enter key, activating the selected option. To return to a previous menu, highlight the carat (^) at the top left corner of the LCD screen and press the **MENU** button.

COND Button

The **COND** button activates the refrigeration system. This provides system cooling for vapor condensing.

VAC Button

The **VAC** button enables the vacuum pump and opens the vacuum brake solenoid (VBS) valve, allowing the system to be evacuated. Vacuum level control options can be programmed from the User Options screen.

*Note: If your vacuum pump is not connected to the IEC plug on the rear of the BenchTop unit, the **VAC** button will not control your pump. See your pump manual for operation instructions.*

AUTO Button

The **AUTO** button will start the refrigeration and vacuum systems in a set sequence. The condenser will first cool to the condenser setpoint. Once the setpoint is achieved, the vacuum pump will energize and begin evacuating the system.

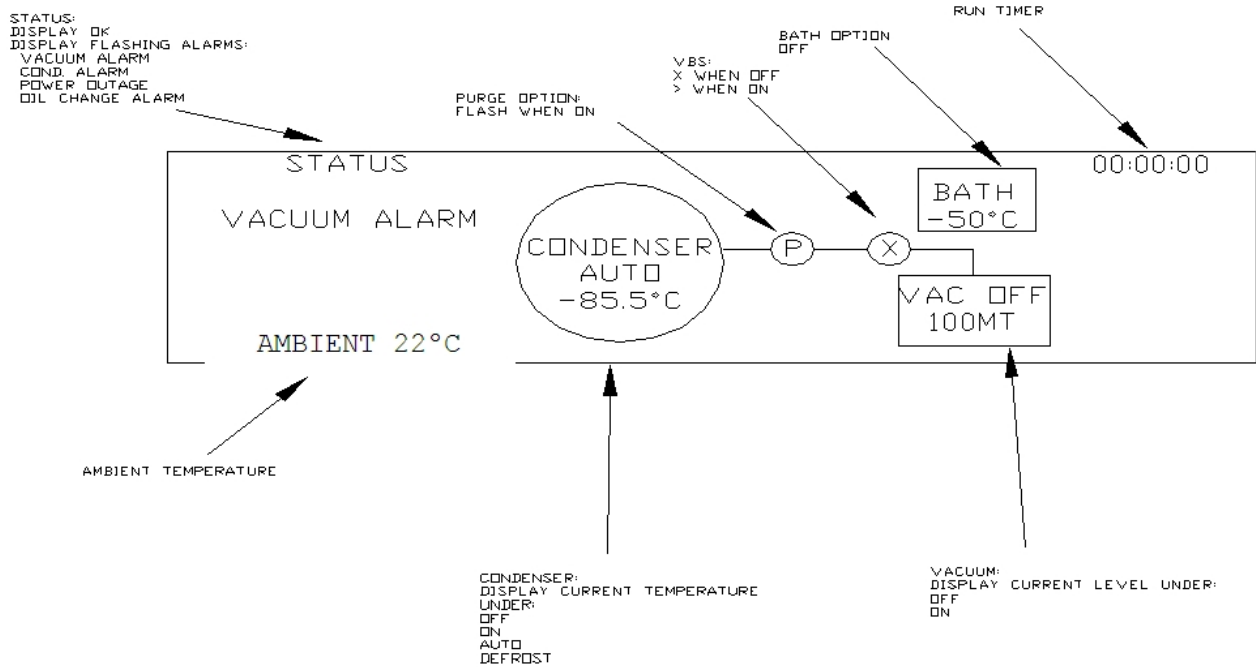
DEFR Button

The **DEFR** button activates the condenser defrost function. Hot gas defrost is directed toward the condenser walls to quickly loosen ice.

Note: Defrost mode is disabled while any other function is on.

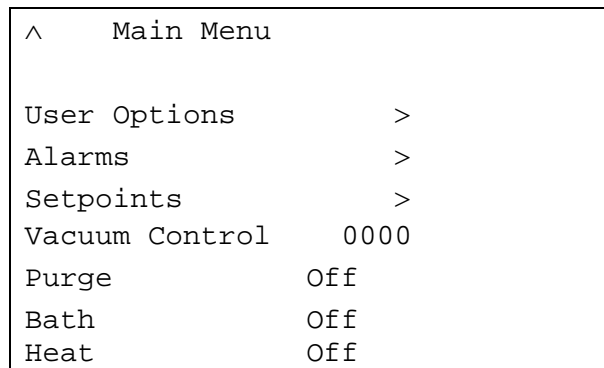
LCD Display Functions

By default, the LCD display shows the system Synoptic screen. The Synoptic screen gives a visual representation of the current operation and status of the unit. Alarm messages, run time and system status are displayed.



Main Menu

Pressing the **MENU** button while on the Synoptic screen displays the Main Menu. If the unit goes idle (no input after three minutes), the system will default back to the Synoptic screen.



Note: Temperature and vacuum control functions are disabled except when the Synoptic screen is displayed. Bath and Heat functions are not available simultaneously.

Navigation within the menu screens is accomplished using the **Up/Down (+/-)** arrow buttons in conjunction with the **MENU** button. The **Up/Down (+/-)** buttons allow you to scroll the cursor to the desired function and allows the value of a setpoint to be changed. The **MENU** button is used to select the desired item.

The \wedge symbol, located in the upper left corner of the screen, returns you to the previous screen. The \gt symbol allows you to go to the next screen within a selected option.

Vacuum Control

The Vacuum Control field displays the current vacuum level control setpoint. To change the setpoint, use the **Up/Down (+/-)** buttons and navigate to the Vacuum Control field. Press and hold the **MENU** button. When prompted, use the **Up/Down (+/-)** buttons to adjust the value. Press **MENU** to enter the new setpoint.

Purge (Optional)

The Purge function controls a VBS valve located between the vacuum pump and the suction port of the unit. This allows high vacuum to be pulled within the volume between the pump and the valve, without submitting the entire unit to high vacuum. The gas ballast can then be opened and the oil cleansed within the pump.

Heat (Optional)

The Heat function gives the ability to add controlled heat to product shelves for enhanced drying. If installed, you can set the temperature of the manifold shelves up to 60 °C.

Note: Optional features will only be displayed if the factory-installed options were purchased with your unit.

User Options Menu

To access the User Options menu, highlight User Options from the Main Menu and press the **MENU** button.

| | | |
|----------|-------------------------|-------|
| \wedge | User Options | |
| | Vacuum Control | VLC |
| | Temperature Units | C |
| | Vacuum Units | Torr |
| | Relay Test | \gt |
| | Vacuum Pump Maintenance | \gt |

Vacuum Control

The Vacuum Control field sets the method used for vacuum level control—None, VLC or Power Save. Setting Vacuum Control to None disables vacuum level control. Setting Vacuum Control to VLC allows gas to bleed into the system through a control valve to maintain the vacuum setpoint. Setting Vacuum Control to Power Save isolates the system and maintains pressure by shutting off power to the vacuum pump and VBS valve. As the system pressure rises, the vacuum pump is re-energized and the system is pulled back down. To avoid rapid cycling of the vacuum pump, the Power Save Deadband should be set to 20 or more.

Temperature Units

The Temperature Units field displays the current setting for indicating temperature values. Temperature can be displayed in either Fahrenheit (°F) or Celsius (°C).

Vacuum Units

The Vacuum Units field displays the current setting for indication of system pressure. System pressure can be displayed in millitorr (mTorr), microbar (μbar) or Pascal (Pa).

Relay Test

The Relay Test screen can be used to troubleshoot electrical or mechanical problems, or to observe active outputs during a freeze-drying run.

```

^ Solid State Relay Output Test
  SSR1          SSR2
  -Vacuum      -Purge
  -Comp2       -Bath
  -Comp1       -Spare
  -Defrost     -Heat
^-SSR1 v-SSR2 Menu-Exit
  
```

To test a relay bank, simply press the **Up or Down (+/-)** button and each of the outputs of the selected relay bank will fire for approximately two seconds each. Relay positions preceded by a highlighted box indicate the output is active.

Vacuum Pump Maintenance

The Vacuum Pump Maintenance screen is used to adjust vacuum maintenance alarm parameters and maintenance time durations.

```

^ Vacuum Pump Maintenance

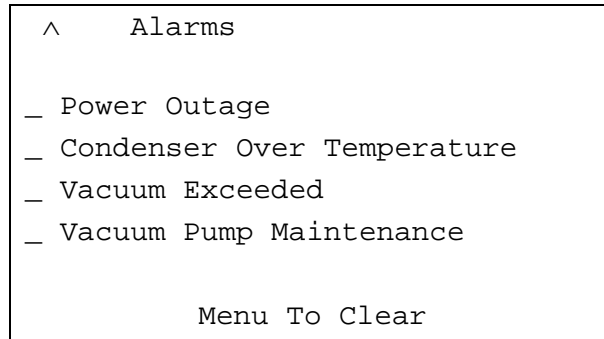
Alarm Setpoint: 240
Current Hours: 0000 min: 007
  
```

The Alarm Setpoint is the number of hours of continuous operation the vacuum pump can run before the vacuum pump maintenance alarm will be generated. This can be used to draw attention to necessary vacuum pump maintenance operations, such as oil changes. The default factory value is 240 hours, but can be changed by pressing and holding the **MENU** button while Alarm Setpoint is highlighted. Once prompted, use the **Up/Down (+/-)** buttons to change this value.

The Current Hours field can be reset at any time by pressing and holding the **MENU** button while Current Hours is highlighted.

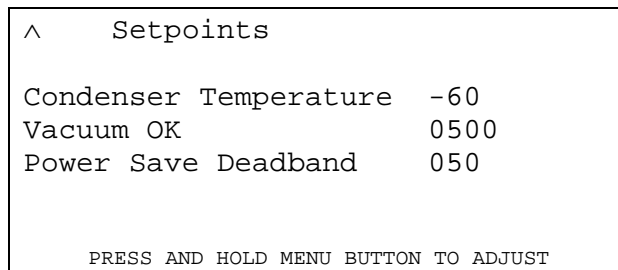
Alarms Menu

The Alarms screen displays all active alarms. A shaded box appears in front of triggered, active alarms. You can also accept and clear triggered alarms from this screen. To clear an alarm, use the **Up/Down (+/-)** buttons to highlight the desired alarm and press the **MENU** button. Before the alarm will clear, the condition that caused the alarm must be resolved.



Setpoints Menu

To edit setpoint values, use the **Up/Down (+/-)** buttons and highlight the desired field. Press and hold the **MENU** button until prompted to adjust the value. Adjust the value with the **Up/Down (+/-)** buttons and press the **MENU** button to enter the setpoint.



Condenser Temperature

The Condenser Temperature setpoint is the temperature at which the vacuum will energize in auto mode. The Condenser Over Temperature alarm is also referenced from this value. The alarm will be triggered when: (1) the condenser temperature rises more than 10 °C above the setpoint temperature after obtaining the setpoint at least once, and (2) the condenser is unable to obtain the setpoint after 30 minutes of starting an automatic cycle.

Vacuum OK

Vacuum OK is the pressure setpoint at which the Vacuum Exceeded alarm will be generated. There is a 30-minute delay in the Vacuum Exceeded alarm to allow flasks to be added to the system without generating this alarm.

Power Save Deadband

The Power Save Deadband is the acceptable amount of pressure rise within the system before the vacuum pump is re-energized. This value is used when Vacuum Control is set to Power Save in the User Options screen.

Maintenance Menu and Factory Options

The Maintenance menus are used for system configuration, calibration and factory setup. Use caution when modifying factory settings.



PROPERTY CAUTION! ENABLING FEATURES THAT ARE NOT INSTALLED MAY CAUSE SERIOUS SYSTEM DAMAGE AND VOID YOUR WARRANTY. ONLY A QUALIFIED SP INDUSTRIES TECHNICIAN SHOULD EDIT VALUES IN THE FACTORY OPTIONS AND INTERSTAGE CONTROL SCREENS.

To access the Maintenance menu, press and hold the **Up/Down (+/-)** buttons simultaneously while on the Main Menu.

| | | |
|---|--------------------|---|
| ^ | Maintenance | |
| | Factory Options | > |
| | Interstage Control | > |
| | Calibration | > |

Factory Options

The Factory Options screen is used to configure the bath, purge, defrost and heat options.

| | | |
|---|-----------------|-----|
| ^ | Factory options | |
| | Bath Option | YES |
| | Purge Option | YES |
| | Hot Gas Defrost | YES |
| | Heat Option | No |
| | ZL Defrost | No |

The optional Heat and Purge components are enabled when set to YES. This results in changes to the Main Menu and Synoptic screens, adding control of these features.

Note: The Heat and Purge options require additional factory-installed components to function. If either option is purchased, the function will be enabled at the factory. If you are unsure whether you purchased the Heat or Purge option, contact SP Industries.

The Hot Gas Defrost option uses a hot gas refrigeration loop to reduce defrost time. Setting the Hot Gas Defrost field to YES enables the hot gas defrost.

Interstage Control

If your unit is equipped with a cascade refrigeration system, you can control the operation of the second compressor from the Interstage Control screen. A cascade system is a safety feature used to protect the refrigeration system from overloading.

| ^ Interstage Control | |
|----------------------|----------|
| Interstage | ON |
| ON -12 | DIFF 025 |
| Current Temp | 1.3 °C |

If the Interstage Control is set to ON, the **COND** or **AUTO** buttons will activate the high-stage compressor. The Interstage Control's Current Temp is monitored until the Interstage ON temperature is reached. Once the Interstage ON temperature is reached, the low-stage compressor is activated.

The DIFF temperature is used to prevent overloading of the high-stage compressor. If the Current Temp exceeds the sum of the Interstage ON temperature and the DIFF value, the low-stage compressor is deactivated. It will reactivate when the Current Temp recovers.

Calibration

The Calibration screen allows modification and control of condenser temperature and vacuum settings. The following procedures require removal of the electrical box cover under the unit's right-side panel.



CAUTION! REMOVAL OF THE ELECTRICAL BOX EXPOSES HIGH-VOLTAGE COMPONENTS. USE EXTREME CAUTION AND FOLLOW ELECTRICAL SAFETY PRACTICES.

| ^ Calibration | |
|------------------------|-----|
| Temperature Span | 070 |
| Temperature Zero 14.5C | 128 |
| Vacuum 760 Torr | 128 |

Condenser Temperature Calibration

To calibrate the condenser temperature, connect a thermocouple temperature simulator to the condenser temperature input. Set the simulator temperature to 25 °C.

The current temperature is displayed on the Calibration screen. Adjust the numerical value of the Temperature Zero as appropriate and press **MENU** to enter the value.

Upon proper adjustment of the Temperature Zero, change the temperature simulator value to -60 °C. Adjust the Temperature Span value so that the temperature displayed equals the calibrator set value. Recheck the value at 25 °C and repeat adjustment if necessary. Check calibration at desired temperature intervals for verification.

Vacuum Value

To adjust the Vacuum value, open the condenser door and adjust the trim pot located on the main Sentry 2.0 module within the electrical box. Set the value to 760 Torr, or current atmospheric pressure. Turn the trim pot clockwise to increase the pressure or counter-clockwise to decrease the pressure.

Once the rough vacuum has been set, connect a reference tube. Adjust the Vacuum value located on the LCD screen up to increase the Vacuum reading. Adjust the Vacuum value down to decrease the Vacuum reading.

Note: SP Industries recommends the use of a Varian Vacuum Reference Tube for vacuum calibration.



PRACTICAL OPERATING TIP. ENSURE THAT THE GROUND WIRE IS FIRMLY CONNECTED BEFORE CALIBRATING ANY VALUES. A LOOSE GROUND WIRE CAN PRODUCE INCONSISTENT AND UNPREDICTABLE RESULTS.



Basic Operation

Getting Started

Prior to operating your freeze dryer, ensure that your condenser is clean and dry. If your unit is equipped with a shelf rack with stoppering, check that the stoppering mechanism is fully retracted before proceeding.

Product Preparation: Flask / Manifold Drying

BenchTop series units include Quickseal valves for processing samples using flasks or other glassware. To prepare samples for freeze-drying in glassware:

1. Fill flasks with your product.³ Do not fill more than half of the flask's total capacity. When using Wide Mouth Flasks, ensure that the filter paper and O-ring are positioned correctly within the flask cover. Snap the cover securely onto the flask.⁴
2. Pre-freeze product samples in a laboratory freezer, dry ice bath or shell bath freezer. Freezing to -40 °C is adequate for most products.⁵

Product Preparation: Drum Manifold Shelf Drying

As an alternative to using flasks, you may use an optional drum manifold and shelves to dry vials of product. To prepare samples for shelf drying:

1. Fill vials or other suitable containers with product.³ Do not fill containers to more than half of their total capacity.
2. Pre-freeze product samples. This can be accomplished directly on the shelves, or in a laboratory freezer, dry ice bath or shell bath freezer. Freezing to -40 °C is adequate for most products.⁵
3. If your unit is equipped with the stoppering option and you are processing product in vials, partially insert a split rubber stopper into each vial.
4. Load samples onto the shelf (or shelves). If you are using product probes, connect the probes to the thermocouple jacks provided.

³ Refer to Appendix B for a list of standard serum vial capacities.

⁴ Wide Mouth Flasks are the most popular glassware type, but other types of flasks, vials and ampoules are available. If you are not certain how to use glassware accessories, contact SP Industries.

⁵ Dry ice methods can freeze a product to approximately -78 °C, while liquid nitrogen (N_{2(lq)}) methods can freeze a product to approximately -190 °C.

Product Preparation: Chamber Drying

BenchTop series freeze dryers can process product in vials or trays directly in the condenser chamber. To process samples in the chamber:

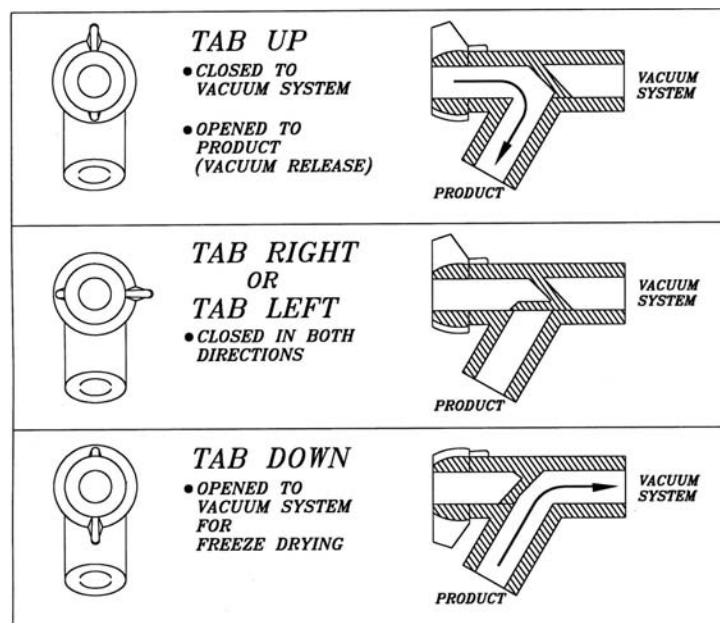
1. Fill tray(s) or vials with your product.⁶ Do not exceed more than half of the container's total capacity.
2. If using a BenchTop 2K or 4K, ensure that the vacuum baffle plate is in the desired position (*i.e.*, position 1 for pre-freezing or position 2 for freeze-drying).
3. If desired, insert product probes into the product. For accurate temperature readings, probe ends should be close to, but not in contact with, the bottom of the container.⁷
4. Close and secure the chamber lid.



PRACTICAL OPERATING TIP. TO MINIMIZE DRYING TIME, ALWAYS FREEZE PRODUCTS IN AS THIN A LAYER AS POSSIBLE. NEVER FILL A CONTAINER TO MORE THAN HALF ITS TOTAL CAPACITY.

Quickseal Valves

Quickseal valves are utilized in many method of drying. They permit the attachment of flasks for *in vitro* freeze-drying. They are also used to break vacuum after a cycle completes. Quickseal valves operate as follows:



Note: Quickseal valves are an integral part of the freeze-drying process and must be maintained as such. For information regarding upkeep and service of Quickseal valves, see the General Maintenance section of this manual.

⁶ Refer to Appendix B for a list of standard serum vial capacities.

⁷ For precise positioning of probes in vials, contact SP Industries about optional MVP Probe Holders.

Operation Instructions

1. Ensure that the condenser is clean and dry before proceeding. Check the drain line for residual moisture, which can cause slow vacuum pump-down. Ensure that the plastic quick-connect drain fitting is not inserted into the drain fitting receptacle on the front of the unit.
2. Pre-freeze the product to a minimum of -40 °C.
3. If you are freeze-drying product on manifold shelves or directly in the chamber and your unit's heat option includes the ability to track product temperature through a product probe, insert the probes. They should be placed close to the bottom of the sample, but not touching it.
Note: Temperature probes can only be used with racks and feed-through adapter plates.
4. Check that all connections and ports are secure. Ensure that Quickseal valves are closed and that the drain plug is removed from the drain fitting.
5. Press the **COND** button to begin cooling the condenser. Wait for the condenser to reach at least -40 °C (*i.e.*, approximately 20 to 30 minutes). Before proceeding, confirm that the condenser temperature status light is green on the LED Wave display and the Synoptic screen displays the condenser temperature at -40 °C or colder.

Alternatively, press the **AUTO** button and allow the system to proceed through the freeze-drying process using the defined settings. If using the **AUTO** function, skip to step 8.

6. Enable vacuum by pressing **VAC**. Allow the system to evacuate the system. Before proceeding, confirm that the vacuum status light is green on the LED Wave display and that the desired vacuum level is displayed on the Synoptic screen.
Note: If your vacuum pump is not connected to the BenchTop unit, the Sentry 2.0 controller will not be able to enable it. Switch on the vacuum manually. See your vacuum pump operator's manual for more information.
7. Add product as appropriate:
 - a. If using a manifold, attach a flask to a Quickseal valve and turn the valve to the open position to begin the freeze-drying process. Allow the vacuum to recover to at least 200 millitorr or your vacuum control setpoint before connecting additional flasks.
 - b. If using a vacuum concentrator, add samples and begin spinning.
Note: For more information, refer to Chapter 6: Cold Trap Condenser.
8. Ensure that critical system parameters (*i.e.*, refrigeration and vacuum) stay within the acceptable ranges. The LED Wave on the control panel provides a quick reference. Periodically check the condenser for ice build-up and defrost as needed.
9. Observe the product to determine when drying is complete. For manifold drying, the process is complete when the outside of the flasks lose condensation and the product appears dry.

For heated shelf and chamber drying, the process is complete when one or more of the following are true:

- a. The condenser approaches its maximum low temperature.
 - b. System vacuum approaches a constant low pressure reading.
 - c. Product temperature is steady and close to shelf temperature (requires use of a product probe).
 - d. Appearance of the product indicates the dryness (*i.e.*, uniformly powdery or fluffy).
 - e. The product probe reads near ambient temperature or the temperature of the heated rack (if equipped with that option).
10. Switch off the vacuum and refrigeration systems. If running an automatic cycle, press the **AUTO** button to disable both systems simultaneously.
 11. For manifold flask drying, close Quickseal valves and remove flasks. If using a vacuum concentrator, turn off the unit and close Quickseal valves. If using a chamber or manifold shelves, break vacuum and remove the product.
 12. If your system is still under vacuum, release vacuum by inserting the drain plug into the drain fitting or by opening a Quickseal valve.
 13. After removing all product, press the **DEFR** button to enable condenser defrost. The defrost function allows ice to be lifted out of the condenser without fully melting. A drain line is connected to a plastic quick-connect fitting, which is then inserted into the drain receptacle on the front of the unit. To open the drain, push the fitting into the drain receptacle. To close the drain line, press the small gray release button on the top of the receptacle. The fitting will pop out.
Note: The quick-connect fitting must be removed prior to freeze-drying, or else the appropriate vacuum pressure will not be achieved.
 14. Once the ice has melted away from the condenser, the ice can be removed. Thoroughly clean and rinse the condenser with a mild detergent or baking soda solution (to neutralize acids).
Note: Do not chip away at the ice, as this may damage the condenser.
 15. The defrost system turns off automatically after an hour. To disable it manually, press the **DEFR** button.

Meltback

Products that have low freezing points are prone to meltback during freeze-drying. Meltback is a term used to describe when the product melts or defrosts before sublimation completes. The following suggestions may help to prevent meltback:

- Decrease the volume and depth of the product.
- Insulate the product container to slow down sublimation caused by an ambient heat source.
- Decrease the amount of product or number of samples connected.
- Verify that the equipment is functioning properly and ensure that room temperature is sufficient to assist the air-cooled refrigeration system.
- Consider diluting the product with water.

For products that dry readily, alternative heat sources such as a heat lamp may be used to expedite the freeze-drying process.

Preventing Glassware Breakage

- Avoid cleaning glassware with a wire brush as metal-to-glass abrasion creates microscopic scratches which can cause breakage.
- Never fill a flask to more than half of its total capacity (*e.g.*, a 600 mL flask has a working capacity of 300 mL).
- When pre-freezing samples in a storage freezer, tilt glassware at a 30° to 45° angle to increase surface area and reduce stress on the glass.
- To prevent glass-to-glass abrasion when placing flasks in glassware washers, do not allow contact with metal racks or other glassware.

Product Dryness End Point

You can clearly observe drying rates during manifold drying since the glassware and product are completely visible. However, since the glassware is exposed to room temperature, environmental moisture may condense and freeze on the cold outer surface of the flask. This is normal.

As the product ice inside the glassware gradually sublimates, the frost on the outside of the glassware slowly recedes and eventually disappears. By this point, approximately 98% of product moisture has been removed. You can assume drying is complete (<1% moisture content) when the exterior of the glassware is at room temperature.

Stoppering (Optional)

The stoppering option applies pressure to partially-inserted stoppers, forcing them into the vials while sealing the product under system vacuum. Stoppering can be operated at atmospheric pressure or under vacuum. Stoppering must be performed with evenly distributed vials of equal height to ensure uniform stoppering force distribution and prevent vial shifting.

Stopper vials only after:

- The product is fully dried.
- The vacuum system is turned off.
- The shelf heat control is disabled.

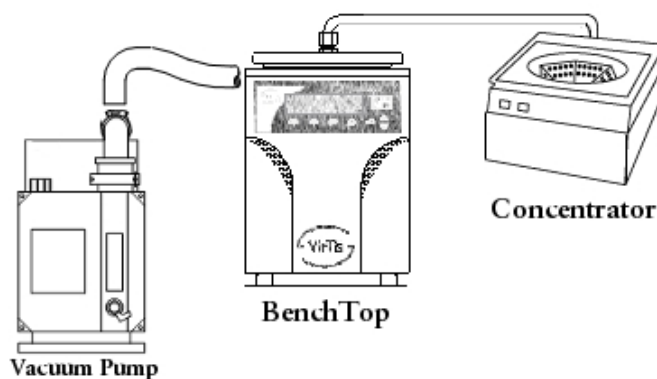
Note: *SP Industries recommends performing a test run to ensure that you are familiar with the stoppering process before adding your product.*

The optional Stoppering system includes a manual stoppering mechanism, and comes with either heated or unheated shelves. For more information, see Chapter 7: Optional Components.

Cold Trap Condenser

Under conditions of high vacuum, vapor pressure of the solvent collecting on the condenser is much lower than that of the product. When vapor molecules leave the product in a vacuum concentrator, they migrate toward low-pressure areas in the condenser. Upon contact with the condenser, the vapors release their heat energy and condense. Whether or not ice forms is dependent on the chemical properties of the solvent. Efficient evaporation rates are dependent on maintaining this vapor pressure differential.

These migrating vapors can be pulled directly into the vacuum pump if condensation does not occur. BenchTop series freeze dryers can be utilized as a Cold Trap Condenser Module to condense and collect vapors being driven out of a product in a vacuum concentrator. Condensing the vapors prevents them from migrating to the vacuum pump and causing damage.



Product Requirements

Before using a Cold Trap Condenser, consider the following:

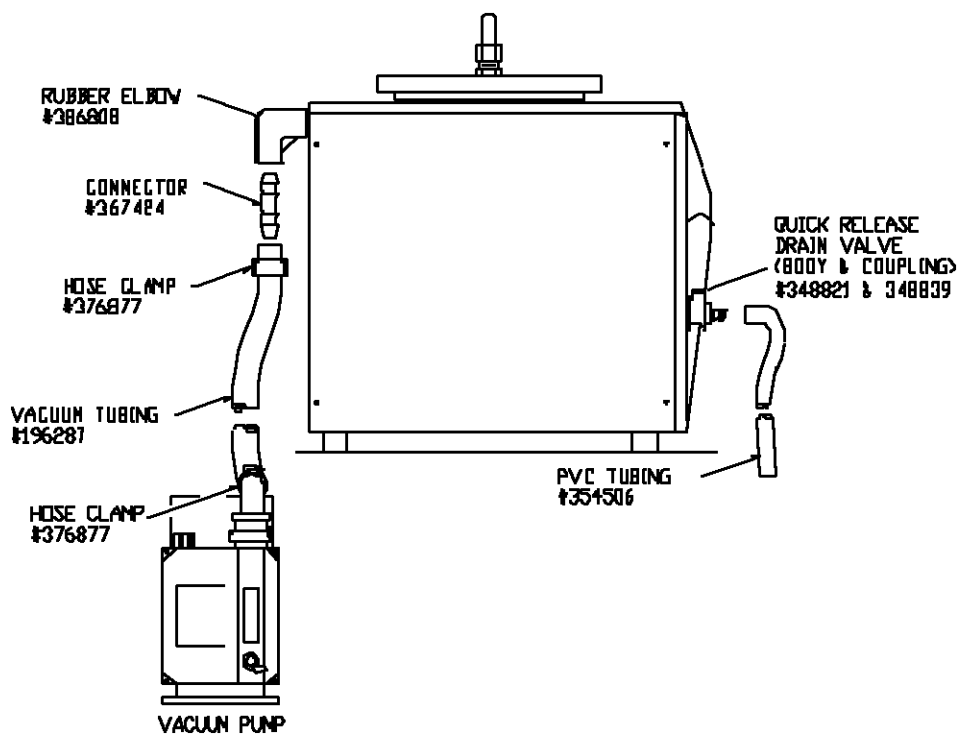
- Freezing and condensing points of solvents.
- Overall volume.
- Solvent properties, such as toxicity.

Note: This process is completely product dependent, so each application will vary in terms of temperature requirements and process time.

Cold Trap Setup

The following instructions will help you utilize your BenchTop as a cold trap to condense vapors from a gel dryer or vacuum concentrator:

1. Position the vacuum concentrator, BenchTop and vacuum pump in a convenient location with sufficient space to easily connect the units.
2. Place the vacuum cover plate (acrylic plate with stainless steel elbow) on the gasket of the BenchTop unit.
3. Connect the BenchTop unit to the vacuum concentrator using $\frac{3}{4}$ -inch tubing.
4. Estimate the length of tubing required to adequately reach the back of the vacuum concentrator without any tension.
5. If necessary cut the tubing to the proper length and secure both ends with tubing clamps.
6. Connect the vacuum pump to your BenchTop unit as described in Chapter 2: Installation and Setup.



Optional Components

The VirTis BenchTop series offers a wide variety of optional components to match a variety of product or process applications.

Manifolds

Many manifolds are available for BenchTop units. Optional manifolds include VirTis 3/4-inch (19 mm) Quickseal valves for attaching a variety of flasks, ampoules or vials. Stainless steel vertical manifolds are available for flask drying, while stainless steel and acrylic drum manifolds are available for the versatility of both flask and shelf rack drying. The top plug on acrylic drum manifolds is removable to allow for installation of an optional stoppering assembly.

Available manifolds include:

- Stainless steel vertical manifolds with 4, 8, 12 or 24 ports.
- Stainless steel drum manifolds with 12 or 18 ports.
- Acrylic drum manifolds with 0, 8 or 12 ports.



For more information about manifolds and current availability, contact SP Industries.

Shelf Racks

VirTis BenchTop units have a variety of shelf racks available for use with the optional stainless steel and acrylic drum manifolds. Racks are available in both heated and unheated configurations. Stoppering racks are also available.

Available rack assemblies include:

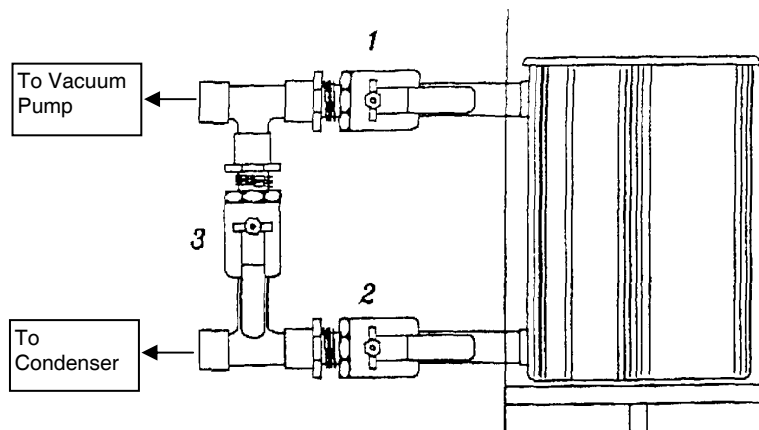
- Stoppering shelf rack with 1, 2, or 3 shelves available with heated or non-heated shelves (for use with acrylic drum manifolds only).
- Bulk processing shelf rack with 3, 4, or 5 shelves available with heated (for use with acrylic drum manifolds only) or non-heated shelves.



For more information about shelf racks and current availability, contact SP Industries.

Liquid Nitrogen Trap

The optional Liquid Nitrogen ($N_{2(liq)}$) Trap protects your vacuum pump from the corrosive properties of acid and alkaline vapors, as well as the low temperatures and organic solvents associated with the freeze-drying process. The trap is available in both 2.8- and 5-liter capacities.



Vacuum bypass assembly with
lever-activated ball valves

To operate a liquid nitrogen trap:

1. Enable the condenser and wait for the system to reach operating temperature.
2. Remove the plastic cover from the stainless steel vessel and add a small amount of liquid nitrogen to the center well. If adding liquid nitrogen from a tank, use adequate ventilation to protect from gaseous vapors.
Note: The liquid nitrogen will expand and bubble when it meets the warm center well. Wear protective eyewear and gloves to avoid contact with skin.
3. Replace cover and wait one minute.
4. Open center well and slowly fill with liquid nitrogen (2.8 or 5 liters, depending on capacity).
5. Open valves 1 and 2, and close valve 3 (see diagram above).
6. Enable the vacuum and wait until vacuum pressure reaches 100 mT.
7. Trap is now in operation. Liquid Nitrogen evaporates rapidly over time and solvent is condensed. Check center well often and refill with liquid nitrogen as needed.

Filter Trap

VirTis filter traps protect your vacuum pump from damaging and corrosive vapors released during freeze-drying. If purchased, the filter trap is located on the side of your freeze dryer, near the vacuum pump.

Disposable Sodasorb and activated charcoal drop-in wire mesh cartridges are available and must be purchased separately.

To install the filter trap:

1. Connect the vacuum pump to the port marked OUT.
2. Connect the rest of the system to the port marked IN.
3. Turn the handle fully counterclockwise to activate the filter trap. The inlet and outlet ports will open.
4. To turn off the filter trap, turn the handle fully clockwise. The inlet and outlet ports will close, breaking vacuum from inside the housing.

Degassing Filter Trap Cartridges

Filter trap cartridges are highly absorbent and may require degassing prior to freeze-drying. If you do not degas filter trap cartridges, vacuum pump-down may take significantly longer. Degassing is also necessary each time you change your filter trap cartridge. Most filters need to be degassed overnight in order to allow proper vacuum to be established.

To degas the filter trap cartridge:

1. Unscrew the clear polypropylene housing from the white plastic top.
2. To begin degassing, enable the vacuum pump, open the gas ballast valve slightly and close all ports.
3. Degassing is complete when vacuum reaches 50 mT, or less. Close the gas ballast.

Note: *Degassing may take between 12 and 24 hours.*

Filter trap cartridges are disposable. Their lifespan depends on frequency of use and the volume of contaminants they absorb. The Sodasorb cartridges contain an "Absorbent Exhaustion Indicator," which turns purple to signify the need for replacement. Full exhaustion is indicated by a deep purple color.

The activated charcoal cartridges contain no absorption limit indicator. Pump oil must be routinely checked for clarity to assess the charcoal's absorbing capacity. Depending on the usage and concentration, changing the charcoal cartridge every three to six months may be sufficient.

BenchTop Cold Plate

The optional BenchTop cold plate can be cooled to -40 °C within 30 minutes when fully insulated and covered. The cold plate will remain lower than -40 °C after the cover is removed. These specifications are based on SP Industries test data from similar units operating with no load.

Initial Setup of the BenchTop Cold Plate

Remove all packing materials carefully and inspect for shipping damage. In the event that shipping damage has occurred, retain all packing material and immediately contact your freight carrier.



PROPERTY CAUTION! THE COLD PLATE IS CONNECTED TO THE BENCHTOP UNIT BY A FLEXIBLE HOSE. THE FLEXIBLE HOSE IS CHARGED WITH REFRIGERANT AND THEREFORE SPECIAL CARE SHOULD BE TAKEN WHEN REMOVING THE COLD PLATE AND BENCHTOP UNIT FROM THE SHIPPING CONTAINERS. THE FLEXIBLE HOSE MAY BE SHAPED OR FORMED WHEN THE UNIT IS AT ROOM TEMPERATURE. DO NOT BEND THE HOSE WHILE AT LOW OPERATING TEMPERATURES

1. Position the BenchTop unit on a stable, flat surface. The cold plate must be fully insulated and have a removable cover (*i.e.*, 1/2-inch thick Styrofoam board or polyurethane foam). This will ensure that the lowest possible temperatures will be achieved. The hose between the plate and the unit is very flexible and can also be inserted into the rear of the unit (approximately 10 inches) so that the length can be adjusted.
2. Verify the outlet you intend to use provides the same voltage and ampacity listed on the serial tag of your unit (*i.e.*, 100-115 Volts, 15 Amps, 60Hz).
3. Verify the shelf thermocouple probe is inserted into the well at the rear of the cold plate, and the thermocouple connector is plugged into the shelf probe receptacle at the rear of the BenchTop cabinet.

Cold Plate Operation

1. Ensure that the power cord is plugged in. Switch the circuit breaker on the rear of the BenchTop cabinet to the “ON” position.
2. Press the **COND** button on the front panel of the BenchTop to start the compressor and begin cooling the cold plate. Wait approximately 30 minutes for the condenser temperature to reach -40 °C. After about an hour, the unit will be capable of achieving lower temperatures (*i.e.*, -50 °C).
3. The cold plate is now ready for use.
Note: When the cold plate is in operation and no product is being added, the cold plate should be covered to prevent frost build-up.
4. When finished, press the **COND** button to disable the refrigeration system and disable cooling to the cold plate.



PRACTICAL OPERATING TIP. THE ONLY BUTTON REQUIRED FOR OPERATING THE COLD PLATE IS THE CONDENSER (**COND**) BUTTON. THE DEFROST, VACUUM AND AUTO BUTTONS DO NOT HAVE A FUNCTION FOR THE COLD PLATE APPLICATION.

General Maintenance

Proper routine maintenance is the key to an efficiently operating unit with minimal downtime. The following section provides instructions on how to maintain your BenchTop series freeze dryer.

Vacuum System

Vacuum Pump

Clean oil is necessary for the best vacuum and overall efficiency of the system. Checking and changing the oil on a consistent basis will greatly extend the life of the vacuum pump.

Check the vacuum pump oil after each freeze-dry cycle by draining a small amount (~100 mL) from the pump drain line. Use a clear container to capture the sample. Oil should be changed as needed.

When visually checking the oil, use the following guidelines:

- Pale yellow or clear vacuum pump oil indicates good condition.
- Dark vacuum pump oil indicates acid contamination.
- Cloudy gray vacuum pump oil indicates water contamination.

Changing Vacuum Pump Oil

Change the oil immediately after shutting down the freeze dryer while the oil is still hot.

1. Protect your hands from the hot oil.
2. Make sure vacuum is released from the system.
3. Remove the top fill plug and open the drain valve located at the bottom of the pump. Drain the contaminated oil into a suitable container.
4. When the oil has completely drained, close the valve and add new oil to the pump while visually checking the sight glass to ensure proper level (near the MAX line). Reinstall the fill plug.
5. If the pump oil is contaminated, operate the vacuum pump for 10 to 15 minutes to flush any residual oil from the system's interior components. Repeat steps 3 and 4 to complete the process.



PROPERTY CAUTION! OIL MUST BE CHECKED AND CHANGED MORE FREQUENTLY IF YOUR PRODUCT CONTAINS CORROSIVE MATERIALS OR ORGANIC SOLVENTS. IN ADDITION, A FILTER TRAP MAY BE INSTALLED TO PROTECT THE VACUUM PUMP.

Scheduling Oil Changes

After clean oil is loaded into the vacuum pump and all necessary connections have been made between the vacuum pump and the freeze dryer, perform a full capacity test cycle.

Have a qualified technician check the oil after the test cycle. If the oil appears dirty, change the oil after every use. If the used oil appears clean, change the oil after the next two uses or cycles. If the oil remains clean after two cycles, change the oil after the next four cycles. Continue to monitor the vacuum pump oil after each cycle until a change of condition is noted or a period of one month has elapsed. If the oil remains clean after several cycles, changing the oil once per month may be sufficient.

Vacuum Tubing and Gaskets

Inspect tubing and gaskets periodically for signs of wear. Check gaskets by removing and inspecting interior surfaces for potential problems. A light coating of vacuum grease on the exterior surfaces will protect gaskets and tubing. Reapply grease as needed.

Condenser Gasket

The condenser gasket should be removed routinely and inspected for cracks.

To check the condenser gasket:

1. Remove the condenser gasket.
2. Take the gasket in both hands and turn it inside out.
3. Inspect the inside of the gasket. This is where you are most likely to find a potential problem.
4. If you find the slightest inconsistency in the surface of the material (*e.g.*, cuts, cracks, dry rot, rippling), replace the gasket as soon as possible.
5. Always install gaskets on a clean, grease-free metal rim. Remove excess grease from the metal using isopropyl alcohol on a fresh paper towel or clean cloth. Clean the gasket with isopropyl alcohol.
6. Once the new or cleaned gasket is installed, apply a very thin coating of high vacuum grease to the outer surface of the gasket only. The gasket should appear moist.

Vacuum Pump Gas Ballast Valve

The gas ballast valve removes some contaminants from the pump oil. During freeze-drying or cold trap use, vapors may bypass the condenser and end up in the vacuum pump. If this occurs, the vapors will degrade the oil causing excessive wear and poor vacuum pressure.

When the ballast is open, it allows a controlled amount of air into the second stage pump cylinder. This reduces the partial pressure, increases the pump's operating temperature and releases the vapors.

Note: Refer to the vacuum pump manual for the location of the gas ballast valve.

Quickseal Valves

VirTis BenchTop units come equipped with Quickseal valves. These valves should be serviced at least once a year.

To service the Quickseal valves:

1. Remove the valves from their ports.
2. Twist and pull the white selector until it is dislodged from the black rubber body.
3. Clean the valves thoroughly with isopropyl alcohol to remove grease and dirt.
4. Inspect each cleaned selector carefully for damage.

Note: Do not use broken flask adaptors, as they can damage the white selector.

5. If scratches or cracks are found, the selector part(s) should be replaced.
6. Apply a thin film of high vacuum grease to acceptable selector(s) and reassemble.

Refrigeration System

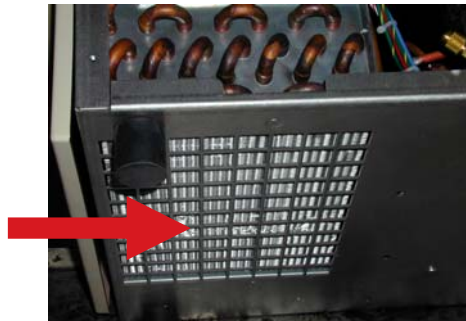
The Air-Cooled Condenser

It is very important to keep the air-cooled condenser clean. This is where high-pressure vapor from the compressor is converted to liquid refrigerant by rejecting the heat gained from the vapor condenser into the ambient air. Reduced airflow over the condenser can result in severely reduced performance and may shorten the life of the compressor.

To maintain the air-cooled condenser and compressor:

1. Do not allow any paper products (e.g., paper towels, Kimwipes®) or cloth to slip underneath the unit. These items will obstruct the airflow.
2. Always maintain at least four inches of clearance on all sides of the unit.
3. Maintain the room temperature at approximately 20 °C. Higher temperatures may result in reduced performance and shortened compressor life.
4. Clean the fins on the bottom of the unit. Dust buildup may cause the unit to malfunction.

Keep these fins clean.



Condenser Chamber

The condenser is fabricated from stainless steel. Under normal use, it can be rinsed and kept clean with a mild detergent. If corrosive materials are being freeze-dried, thoroughly clean and rinse all parts of the freeze dryer that come in contact with product vapor. This prevents build-up of corrosive materials, which protects the freeze dryer and subsequent product loads. Add baking soda or a mild buffering agent to the rinse water to help neutralize acidic residue. A siphon type squeeze bottle can be used to direct the rinse spray into difficult to reach areas.

For more information, refer to Appendix C: Stainless Steel Cleaners.

Acrylic Parts

Clean acrylic covers and manifolds with a mild detergent. Use a soft cloth or Kimwipes® to avoid scratching the acrylic.

The following table may be used as a general guide to acrylic's chemical resistances.

| Chemical | Code | Chemical | Code | Chemical | Code |
|---------------------------------|------|---|------|--|------|
| Acetic Acid (5%) | R | Ethyl Alcohol (50%) | LR | Nitric Acid (10%) | R |
| Acetic Acid (Glacial) | N | Ethyl Alcohol (95%) | N | Nitric Acid (40%) | LR |
| Acetic Anhydride | LR | Ethylene Dichloride | N | Nitric Acid (Conc.) | N |
| Acetone | N | Ethylene Glycol | R | Oleic Acid | R |
| Acetonitrile | N | 2-Ethylhexyl Sebacate | R | Olive Oil | R |
| Ammonium Chloride (Saturated) | R | Formaldehyde (40%) | R | Phenol Solution (5%) | N |
| Ammonium Hydroxide (10%) | R | Gasoline (Regular, Leaded) | LR | Soap Solution (Ivory) | R |
| Ammonium Hydroxide (Conc.) | R | Glycerine | R | Sodium Carbonate (2%) | R |
| Aniline | N | Heptane | R | Sodium Carbonate (20%) | R |
| Battery Acid | R | Hexane (Commercial Grade) | R | Sodium Chloride (10%) | R |
| Benzene | N | Hydrochloric Acid | N | Sodium Hydroxide (1%) | R |
| Benzyl Alcohol | N | Hydrofluoric Acid (40%) | R | Sodium Hydroxide (10%) | R |
| Butyl Acetate | N | Hydrogen Peroxide (3%) | R | Sodium Hydroxide (60%) | R |
| Calcium Chloride (Sat.) | R | Hydrogen Peroxide (28%) | N | Sodium Hypochlorite (5%) | R |
| Calcium Hypochlorite | R | Isooctane | R | Sulfuric Acid (3%) | R |
| Carbon Tetrachloride | N | Isopropyl Alcohol | LR | Sulfuric Acid (30%) | R |
| Chloroform | N | Kerosene | R | Sulfuric Acid (Conc.) | N |
| Chromic Acid (40%) | N | Lacquer Thinner | N | Toluene | N |
| Citric Acid (10%) | R | Methyl Alcohol (50%) | LR | Transformer Oil | R |
| Cottonseed Oil (Edible) | R | Methyl Alcohol (100%) | N | Trichloroethylene | N |
| Detergent Solution (Heavy Duty) | R | Methyl Ethyl Ketone (MEK) | N | Turpentine | LR |
| Diesel Oil | R | Methylene Chloride | N | Water (Distilled) | R |
| Diethyl Ether | N | Mineral Oil | R | Xylene | N |
| Dimethyl Formamide | N | Naphtha (VM&P) | R | Trifluoroacetic Acid | N |
| Diocetyl Phthalate | N | Codes: R = Resistant (withstands long periods of exposure at temperatures up to 50 °C) | | LR = Limited Resistance (withstands short periods of exposure at room temperature) N = Not Resistant (immediate damage may occur upon exposure) | |
| Ethyl Acetate | N | | | | |



CAUTION! DO NOT USE ORGANIC SOLVENTS OR ABRASIVE CLEANERS.

Appendix A: Troubleshooting

Vacuum Problems

Remember that maximum vacuum is only achievable with a clean, dry system. Initial vacuum will be affected by the amount of product loaded on the unit. For efficient freeze-drying, vacuum should be below 200 mT. When using Quickseal valves, product may need to be introduced in stages to keep the vacuum below acceptable levels.

Consider the following when attempting to pinpoint vacuum-related problems:

- Was the condenser defrosted and drained after the last run?
- Is the chamber clean and dry?
- Is the drain fitting inserted into the drain receptacle? If so, it must be removed.
- Are all accessory ports, valves and filters closed tightly? Check all connections for integrity.
- Is the door gasket clean and properly greased?
- Are all Quickseal valves in the closed position?
- Are the Quickseal valves in good condition? Is a light coating of vacuum grease visible on the valve plug and on the stainless steel valve port?
- Is the vacuum pump oil clean and at the proper level? Check sight glass.
- Is the condenser maintaining the proper temperature? If the temperature rises due to refrigeration problems, moisture may be leaving the condenser walls and migrating to the vacuum pump, causing poor vacuum.

If none of the above applies, try to isolate the problem by removing the manifold or the vacuum cover plate and placing a stopper in the vacuum intake tube at the bottom of the condenser. This will seal the vacuum tube. When the vacuum pump is operated, check the components between the pump and the end of the tube.

If you get a vacuum reading below 500 millitorr within one (1) minute, you can rule out any problem with the pump, vacuum probe and vacuum tubing connections. Recheck the manifold and gasket for joint separations and dirty or rough surfaces.

If you do not get a normal vacuum reading on the front panel display or vacuum gauge within one (1) minute, start checking components individually.

If none of these suggestions eliminate the vacuum problem:

1. Change and flush the oil in vacuum pump.
2. Disassemble all vacuum tubing connections, clean with a mild alcohol (such as methanol), apply a light coating of vacuum grease and reassemble.
3. Check all threaded and welded connections and seal with vacuum sealant.

If poor vacuum persists, or if testing vacuum with a calibrated gauge indicates a good vacuum reading, the problem is likely related to the vacuum probe. Replace the vacuum probe tube and retest.

Note: *If this does not resolve your vacuum problem, contact SP Industries for assistance.*

Product Melting

The most common reason for product melting is poor vacuum. Adequate vacuum is required to keep products in a frozen state. Check for a vacuum leak or possible restrictions in the vacuum hose as described in the previous section.

If the unit is functioning properly but the product is still melting, the product's freezing point (eutectic temperature) may be too low to remain in a frozen state. Retest using a water sample to determine if a mechanical or product-related problem exists.

Appendix B: Serum Vial Capacities

| VirTis Part Number | Height (mm) ⁸ | Body OD (mm) | Capacity (mL) |
|----------------------|--------------------------|--------------|---------------|
| 179077 ⁹ | 35 | 18 | 2 |
| 179275 ¹⁰ | 85 | 12 | 2 |
| 179101 ⁹ | 40 | 22 | 6 |
| 179085 | 47 | 23 | 5 |
| 179143 ⁹ | 50 | 23 | 10 |
| 179135 | 54 | 26 | 10 |
| 178855 ⁹ | 62 | 28 | 20 |
| 178830 | 58 | 33 | 20 |
| 178897 | 63 | 37 | 30 |
| 178921 | 73 | 43 | 50 |
| 178954 | 95 | 52 | 100 |
| 178988 | 107 | 54 | 125 |

⁸ Add 8mm for partially inserted stopper.

⁹ Thin Wall.

¹⁰ Special Stoppering Ampoule (add 10mm for partially inserted stopper).

Appendix C: Stainless Steel Cleaners

The following table lists possible problem situations and suggests corrective actions. For additional information, contact SP Industries.

| Cleaning Required | Cleaning Agent ¹¹ | Application Method ¹² | Effect on Finish |
|---|---|---|--|
| HEAT TINT OR HEAVY DISCOLORATION. | Penny-Brite or Copper-Brite | Use in direction of polish lines on No.4 (polished) finish. Wipe with dry cloth. | May scratch No.2 (mill) and Nos.7 and 8 (polished) finishes. |
| | Paste Nu-Steel, DuBois Temp, Tarnite, or Kelox | Rub with dry cloth or stainless steel wool. | Use in direction of polish lines on No.4 (polished) finish. May scratch No.2 (mill) and No. 7 and 8 (polished) finishes. |
| | Revere Stainless Steel Cleaner, Take-Off, or AC-60 | Apply with damp sponge or cloth. | Use in direction of polish lines on No.4 (polished) finish. May scratch No.2 (mill) and Nos.7 and 8 (polished) finishes. |
| | Allen Polish, Steel Bright, Wyandotte, Bob-O, Zud, Dubrite, or Prepare Dex | Rub with a damp cloth. | Use in direction of polish lines on No.4 (polished) finish. May scratch No.2 (mill) and Nos.7 and 8 (polished) finishes. |
| TENACIOUS DEPOSITS, RUSTY DISCOLORATIONS, INDUSTRIAL ATMOSPHERIC STAINS. | Oakite No.33, Dilac, Texo 12, Texo N.Y., Flash-Klenz, Caddy Cleaner, Turco Scale 4368, or Permag 57 | Swab and soak with clean cloth. Allow to stand 15 minutes or more, per directions on package. Then rinse and dry. | Satisfactory for use on all finishes. |
| HARD WATER SPOTS AND SCALE. | Vinegar | Swab or wipe with cloth. Rinse with water and dry. | Satisfactory for use on all finishes. |
| | Dilac, Oakite No.33, Texo 12, Texo N.Y | Swab with cloth or soak. Let stand 10-15 minutes. Always follow with neutralizer rinse, and dry. | Satisfactory for use on all finishes. Effective on tenacious deposits or where scale has built up. |

¹¹ Use of proprietary names is only intended to indicate a type of cleaner, and does not constitute any endorsement (nor does omission of any proprietary name imply its inadequacy). All products should be used in strict accordance with instructions and warnings on the product package.

¹² In all applications, a stainless steel wool, sponge, fibrous brush, or pad is recommended. Avoid use of ordinary steel wool or steel brushes for scouring stainless steel.



Appendix D: Spare Parts List

Refer to the following list for commonly used parts.

| 2K | 4K | 6K | Part # | Description |
|----|----|----|--------|--|
| X | X | X | 153650 | Vacuum probe tube 10324pt. |
| | X | X | 154328 | 12-inch gasket for condenser |
| X | X | X | 171884 | ½-inch Quickseal valves |
| X | X | X | 171926 | ¾-inch Quickseal valves |
| X | X | X | 185181 | One gallon can of Vacuum Pump Oil |
| X | X | X | 185207 | Two gallon can of Vacuum Pump Oil |
| X | X | X | 250365 | Sodasorb cartridge for acids |
| X | X | X | 250373 | Activated Charcoal Cartridge for organic solvents |
| X | X | X | 253013 | Oil Mist Eliminator, for 37 LPM Vacuum Pumps |
| X | X | X | 414203 | Oil Mist Eliminator, for 65 LPM Vacuum Pumps |
| X | X | X | 266783 | Molecular Sieve Cartridge for water vapor |
| X | X | X | 272310 | Free Standing Filter Trap |
| X | X | X | 277228 | One liter of synthetic oil |
| X | X | X | 354506 | Drain hose - clear tubing - 36 inches recommended |
| X | X | X | 356808 | 90-degree rubber elbow for vacuum port connection to vacuum pump tubing. |
| X | X | X | 357699 | High Vacuum grease (10 each, .5 oz tubes) |
| | | X | 364604 | Circuit breaker 10 Amp (ES) |
| X | | | 366658 | Oil Separator (XL) |
| X | X | X | 367424 | Plastic vacuum hose connector |
| X | | | 372336 | Compressor 1/3 HP 115 V 60 Hz (ES) |
| | | X | 372343 | Compressor 1/3 HP 208/230 V 60Hz (ES) |
| X | X | X | 374231 | Neoprene foot for BenchTop |
| X | | | 385282 | 9-inch gasket for condenser |
| X | X | X | 398701 | Fan 7.5-inch diameter |
| | X | X | 401579 | Compressor 3/8 HP (EL) |
| X | X | X | 406208 | Needle valve for vacuum level control |
| X | X | X | 381567 | Acrylic manifold |
| X | X | X | 387811 | Manifold tapered plug |

Note: Contact SP Industries for pricing and availability.

SP Industries
3538 Main Street
Stone Ridge, NY 12484 USA
www.SPIndustries.com
(800) 824-0400
(845) 687-0071