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BAF_S04_LABCONCO freeze dryer

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Protocol status: Working

We use this protocol and it's working

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Abstract

General operation of the Lyophilizer unit.

Guidelines

All samples must be frozen PRIOR to putting in the freeze dryer for the sample to dry correctly

Safety warnings

! Alerts: A number of events may occur during a lyophilization procedure that will cause an alert to be displayed on the Freeze Dryer touch screen. An audible alarm (beeper) will also be sounded that will automatically be muted after one minute. The specific alert type can be identified by observing the message box on the display. The alert message box and audible alarm can be dismissed by pressing the “Back” button on the alert message box. The following conditions will initiate an alert.

Power Fail

If a power failure occurs during a Freeze Dry process, the vacuum control valve will allow air to bleed into the Freeze Dryer System. If the failure is of a short duration and the collector does not warm up above -30°C, when power is restored the Freeze Dryer will restart and resume operation of the refrigeration and vacuum systems. If the power failure lasts for a longer duration and the collector warms above safe limits, when the power is restored, the Freeze Dryer will not automatically restart. This prevents melted sample from being drawn into the collector and prevents liquid from being drawn into the vacuum pump. When power is restored, the POWER FAIL alert message will be displayed on the touch screen and the audible alarm will sound.

Line Voltage Range

If the voltage supplied to the Freeze Dryer varies beyond allowable limits, the LINE VOLTAGE RANGE alert will be activated. The high and low alarm points are preset at the factory to correspond to the normal allowable voltage variations based on the nominal voltage specified for the freeze dryer. Some models may be operated outside the normal voltage limits (see Appendix C). If necessary, the high and low LINE VOLTAGE OFFSETS may be adjusted from the Settings / Maintenance screen.

Collector Temp

After the collector temperature has cooled to below -40°C, an alert will be activated if the collector temperature rises above -40°C for more than 5 minutes.

Collector Moisture

If there is liquid in the collector chamber and either the COLLECTOR or VACUUM start button is pressed, neither function will start and an alert will be activated. This feature prevents the inadvertent start of the vacuum pump when liquid is in the collector and thereby can extend the life of the vacuum pump. A few drops of liquid may remain in the drain hose after draining. When the vacuum pump is started these drops may be sucked into the collector chamber. This is normal.

Service Vacuum Pump

The vacuum pump normally plugs into the vacuum pump electrical receptacle on the back of the Freeze Dryer. When the Freeze Dryer has accumulated a total of



1000 operating hours, an alert will be activated. It may be necessary to service the vacuum pump more frequently than every 1000 hours depending on the operation of the Freeze Dryer. The pump oil should be regularly monitored to verify that it is clean. The “Set hours between oil change” can be reprogrammed from the Settings / Maintenance screen for any value from 0 to 9999 hours.

Vacuum Leak

If the vacuum level in the freeze dryer has not reached at least 5 mbar within 30 minutes of starting the vacuum, an alert will be activated and the vacuum pump will be turned off (if it is plugged into the Freeze Dryer vacuum pump outlet).

Vacuum Set Point

If the system is unable to achieve the vacuum set point (during Manual or Program mode), an alert will be activated. This alert will be activated if the vacuum level is not within 0.500 mbar of the vacuum set point in 20 minutes.

Before start

THIS CHECKLIST SHOULD BE FOLLOWED PRIOR TO EACH USE:

1. remove the accessory drying chamber or manifold from the collector chamber lid and using a soft, lint free paper towel, wipe the port gasket(s) and sealing surfaces of the drying chamber/manifold and collector chamber lid to remove any dirt or contaminants that could cause a vacuum leak.
2. Ensure that the collector chamber and drain line are free of water. Place the drain hose in a suitable container to collect the condensate from the collector chamber. Insert the quick connect drain fitting into the quick connect drain coupling located on the left hand side of the Freeze Dryer. **Note: Freeze Dryer (Collector or Vacuum) will not start if moisture is detected in the drain line.**
3. after completely draining the system, disconnect the quick connect drain fitting from the quick connect drain coupling.
4. wipe the interior of the collector chamber with a paper towel to remove any remaining moisture.
5. reinstall the drying chamber or manifold on the collector chamber lid.
6. check that each sample valve is closed or in the VENT position.
7. start COLLECTOR and allow the refrigeration system to reach its specified operating temperature.
 - if using auto start up: go to home screen and press AUTO --> START. collector starts immediately, vacuum starts once collector reaches -40°C
 - if using manual star up: go to home screen, press COLLECTOR --> START. when collector reaches -40°C, manually press VACUUM --> START
9. attach a pre-frozen sample to sample valve on drying chamber or manifold.



Checklist before starting - from User Manual

- 1 Remove the accessory drying chamber or manifold from the collector chamber lid and using a soft, lint-free cloth or paper towel, wipe the port gasket(s) and sealing surfaces of the drying chamber/manifold and collector chamber lid to remove any dirt or contaminants that could cause a vacuum leak.
- 2 Ensure that the collector chamber and drain line are free of water. Place the drain hose in a suitable container to collect the condensate from the collector chamber. Insert the quick connect drain fitting into the quick connect drain coupling located on the left side of the Freeze Dryer. Note: Freeze Dryer (Collector or Vacuum) will not start if moisture is detected in the drain line.
- 3 After completely draining the system, disconnect the quick connect drain fitting from the quick connect drain coupling.
- 4 Wipe the interior of the collector chamber with a soft cloth or paper towel to remove any remaining moisture. Reinstall the drying chamber or manifold on the collector chamber lid. Note: Vacuum grease is NOT required on the drying or collector chamber lid gasket to obtain a proper vacuum seal.
- 5 Check that each sample valve is closed or in the “vent” position.

Turn on the lyophilizer

- 6 Go to the Home screen, for the auto-start procedure click on AUTO and then press START. The collector starts cooling down, and once temperature reaches -40 C, the VACCUM pump will start.
Wait until temperature reaches -80 C temperature, and pressure reaches 0.001 mbar. Depending on sample type these parameters should be adjusted, refer to User Manual.

Add samples

- 7 MAKE SURE ALL SAMPLES ARE PRE-FROZEN
- 8 Connect a pre-frozen sample to a sample valve on the drying chamber or manifold using an adapter. Turn the plastic valve knob to the “VACUUM” position to open the valve, which connects the attached sample to system vacuum. The bevel on the knob should be positioned



toward the sample port to apply vacuum to the sample.

- 9 Before adding another sample, allow system vacuum to return to 0.133 mbar or lower. Any combination of valves and sample sizes may be utilized at one time provided that the system vacuum and collector temperature remain sufficiently low to prevent melting of the frozen sample.
- 10 When all the frost has disappeared from the outer surface of the sample container and no cold spots can be detected by handling the container, the sample is nearly dry. To be certain of low final moisture content, dry the sample for several hours past this point.
- 11 To remove a container after drying is complete, turn the plastic knob on the valve to the "VENT" position, which closes the valve and vents the container. Should back-filling with an inert gas be required, connect the gas supply line to the vent port on the valve before turning the plastic knob on the valve to vent position. The sample container may now be removed. In the vent position the bevel on the valve knob should point away from the sample port
- 12 Ampules may be flame sealed while connected to a valve by using a sealing torch. Care must be taken not to burn the valve. An insulation material placed between the valve and the torch is recommended.

Shut down

- 13 At the end of a run or when a sufficient amount of condensate accumulates on the collector coil to obstruct the flow of vapor to the collector chamber, the Freeze Dryer should be defrosted.
Go to Home screen, press VACUUM, then STOP to turn the vacuum pump OFF. Then release system vacuum by turning the plastic knob on a valve (that has no sample attached) to the open position or opening the vent valve on Tray Dryer or Clear Chamber. Press COLLECTOR, then STOP to turn the refrigeration system OFF.

Defrost the collector coil

- 14 The hot gas defrost function can be used to speed up the defrosting process. Defrost can be activated as follows: Go to Home screen, press COLLECTOR button, press Defrost Options, then press START.
- 15 To turn off Defrost: Go to Home screen, press DEFROST button, press STOP. The defrost function will turn off automatically if the collector temperature reaches +60°C or if defrost has been running for 2 hours.



- 16 If rapid defrost is desired, pour warm water over the collector coil. Do not allow the liquid to enter the vacuum port on the upper rear wall of the chamber.
- 17 Place the drain hose in a suitable container to collect the condensate from the collector chamber. Insert the quick connect drain fitting into the quick connect drain coupling located on the left hand side of the unit.
- 18 Flush the collector chamber with water and wipe chamber dry
- 19 Disconnect the quick connect drain fitting from the quick connect drain coupling

Export data log file

- 20 Data Log Files can be copied (exported) to a USB flash drive via the USB port on the left side of the Freeze Dryer. The file will be exported as a “comma separated values” file (.csv file extension), which can be easily opened with a spreadsheet application program for data analysis and graphing. To export a file:
 - 20.1 Insert USB flash drive into the Freeze Dryer USB port --> Go to Data Logging screen --> Select the file that you want to export by pressing the Data Log File name --> Press the Export button

Protocol references

FreeZone Benchtop Freeze Dryers (7343000 Rev K) - LABCONCO User Manual.