

Lyophilization (Freeze Drying)

version 16.3

Starting the Shell Freezer:



1. Turn on the **PUMP** and confirm that a steady flow of [ethanol](#) enters the bath from the distribution ports. There should be a sufficient flowrate to go halfway up the belts. **Caution: At no time should the unit be operated if "suction sounds" are heard. Such a condition would indicate a shortage of ethanol in the reservoir. Do not run the pump dry as damage will result.**
2. Turn on the refrigeration with the **COOL** switch. In approximately 5 minutes, the temperature of alcohol reaching the belts will be -20°C and shell freezing can begin. **Caution: Do not operate the refrigeration without the pump.**

3. Start the belts with the **DRIVE** switch and adjust the speed as desired. Note that the drive can be stopped to add or remove flasks from the bath, and restarted.

Note: The tilt has been disabled.

Filling Flasks & Freezing Samples:

Caution: Keep the volume of liquid under the lip when tilted on its side.



1. Add the desired amount of slurry to each flask (about 60 mL) being careful to not contaminate the filter at mouth of the flask.
2. Place the sample carefully on the turning belt.
3. Freeze samples in the shell freezer
4. When frozen, remove the sample from the belt with the tongs to place on the freeze dryer.

Note: Read the following link to ascertain optimum shelling.

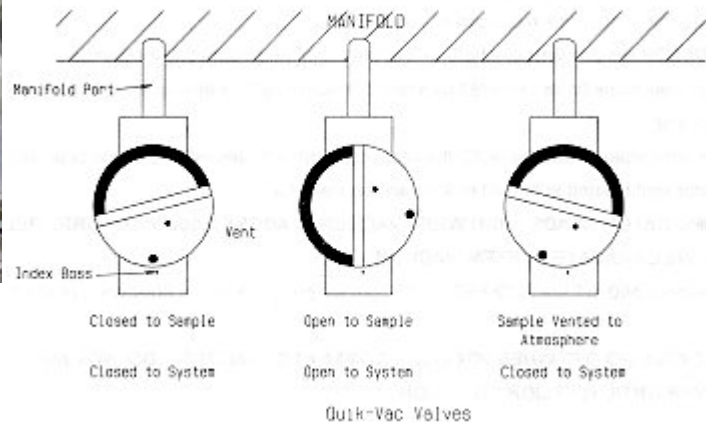
[What is optimum "shelling"?](#)

Startup of Freeze Dryer:

Note: There is a 2 second delay when pushing the START/STOP key pads. The function is activated when the microprocessor makes an audible signal. This delay helps to prevent the unit from being accidentally activated. (ie. The button must be held until it beeps.) The DEFROST key pad no longer works.



1. Check that the oil level in the vacuum pump.
2. Turn on main power switch.
3. Check Ambient Temperature. If ambient temperature exceeds 28°C, DO NOT RUN experiment. If the freeze dryer has not yet been operated, ambient temperature can be obtained from the condenser temperature display of the freeze dryer.
4. Make sure all Quik-Vac Valves are in the closed position.



5. Push the START/STOP key pad until you hear an audible signal (approximately 2 seconds)
6. Condenser compressor turns on as indicated by a green indicator light located to the left of the Temperature indicator.
7. When condenser temperature reaches -40°C, the vacuum pump activates within 5 seconds as indicated by a green indicator light located to the left of the Vacuum indicator. Under normal conditions, the temperature will reach -40°C in about 3-5 minutes. **Caution: If -40°C is not reached within 10 minutes, an alarm will sound and the OUT OF RANGE red TEMP light will be on. This indicates the temperature is too high.**

****Note: Vacuum indicator reads 3HHH when vacuum is above 3,000 millitorr. Below 3,000**

millitorr it will indicate system vacuum.



8. When vacuum reaches 500 mT the ADD PRODUCT light will go on. Add one pre-frozen product flask to the manifold.

Caution: If vacuum does not reach 500 mT within 10 minutes of activation of the vacuum pump an alarm will sound and the OUT OF RANGE red VACUUM light will be on. This indicates the pressure is too high.

****Note:** Add light will go off when product flask is added. DO NOT add another flask until add light goes on again.

****Note:** Only product that is completely frozen should be added to the freeze dryer.

9. Continue to add product flasks one at a time to a maximum of 6 flasks, 3 on a side.

10. Use the balance to the left of the freeze dryer to measure sample weights before and during the experiment.

Caution: If voltage light comes on and remains on, this indicates an inadequate power supply.

System Defrost and Shut Off

****Note:** All product must be removed from unit before shut down.

1. Push the start/stop button until it beeps to stop the freeze dryer.
2. The VACUUM indicator light will go off, indicating the vacuum is no longer operational. Remove the plexiglass cap from the manifold. Be careful not to allow it to hit the ground when the vacuum turns off.
3. Turn off the main power switch and remove the manifold.