

# Lyophilization (Freeze Drying)

version 03.1

## 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 entirely cover 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  $-20^{\circ}\text{C}$  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.

4. Adjust the tilt of the belt platten with the knob on the right front of the shell freezer to the desired angle as in step 3 below.

## Filling Flasks:

**Caution: Never fill flasks to more than half of total volume**

The Quick-Dryflasks ,QD-75, that are used in this experiment have a total volume of 75 ml, therefore never fill with more than 37 ml of slurry.



1. Add the desired amount of slurry to each QD-75 flask being careful to not

contaminate the filter at mouth of the flask.

2. Stopper each flask with the provided silicone stoppers.

3. Freeze samples in the shell freezer, adjusting the angle of the belt platten to achieve optimum "shelling". Note that samples up to 12 ml of slurry can be "shelled" in the QD-75 flasks without the need to tilt the platten.

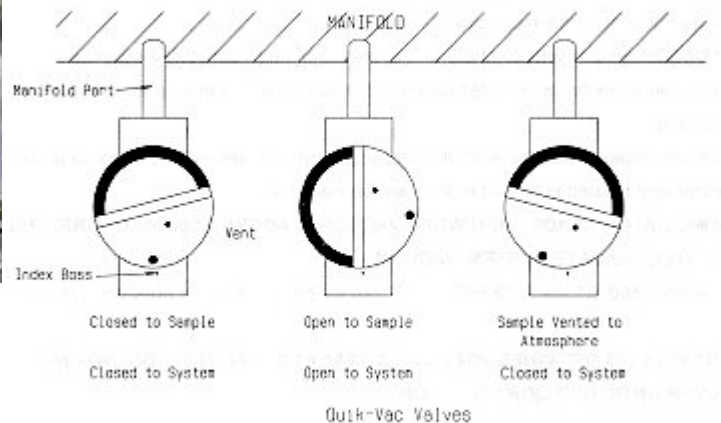
[What is optimum "shelling"?](#)

## Startup of Freeze Dryer:

Note: There is a 2 second delay when pushing the START/STOP and DEFROST 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.)



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.

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

## System Defrost and Shut Off

1. Press the DEFROST button until you hear the audible signal and DEFROST light is on.

**\*\*Note:** All product must be removed from unit before defrosting.

2. The defrost valve is turned on automatically and the system will break vacuum. The vacuum pump will be automatically turned off in the correct sequence.

3. The VACUUM indicator light will go off, indicating the vacuum is no longer operational.

4. The TEMPERATURE indicator remains on until the system reaches +40°C, then the compressor will be automatically turned off.

5. If the temperature reduces rapidly, the defrost cycle will reactivate in 3 minutes.

6. This cycle continues until there is no longer ice on the condenser.

7. When complete, the DEFROST light will go off and the system will shut down automatically.

**\*\*Note:** The amount of time between the beginning of the defrost cycle and the time when the condenser/ice bond breaks is short.

8. When the DEFROST light goes off, turn off the main power switch and collect the water from the condensate drain pan at the rear of the freeze dryer.