LABCONCO 2.5 L Lyophilizer

Explainer

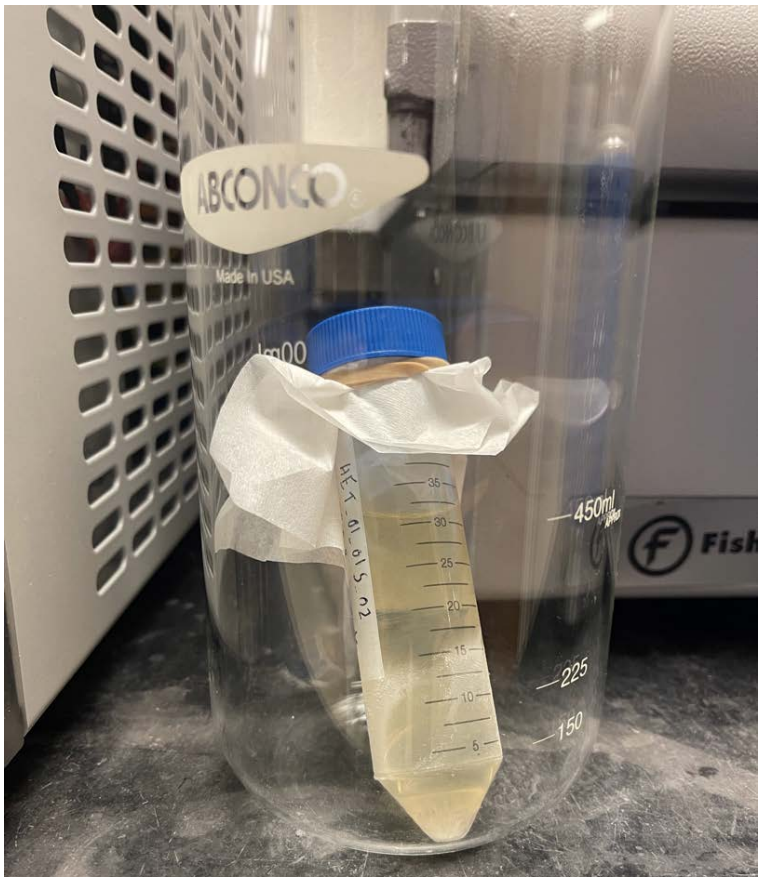
Lyophilizing is powerful as a method to evaporate difficult, high boiling point solvents from samples. This instrument, often called a freeze dryer, uses a frozen sample and sublimes the solid solvent off of the sample at high vacuum. The samples are kept frozen during the process not by active freezing from a freezer but by energy loss through sublimation. Solvents with high boiling points include water, toluene, dimethyl sulfoxide, and dimethyl formamide where. This serves as a substitute for a high vac setup using a schlenk line but often takes much longer than that does.

Running a Sample

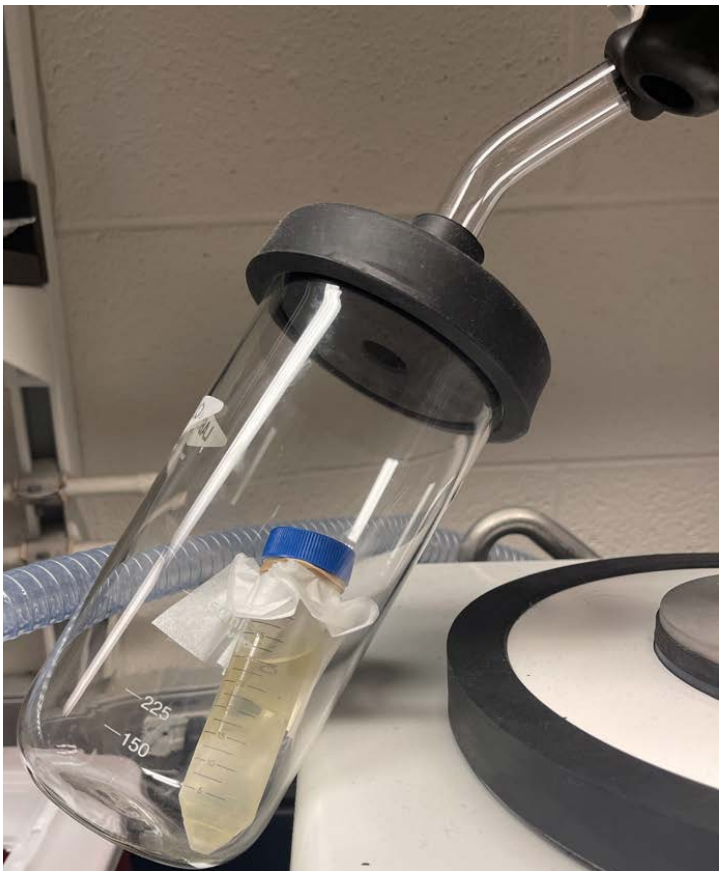
1. Start with a sample that has a high boiling point liquid in it (usually a boiling point higher than 100 °C). Transfer the sample into centrifuge tubes, multiple if necessary, and fully submerge the tube in liquid nitrogen. Wait 20-30 minutes or completely frozen (this time will change depending on how large the sample is).

2. Before using the instrument, make sure the basin (under the metal pole) is clean and clear of any liquid. On the instrument, turn on the lyophilizer cooling. This is done by clicking on the red temperature under ‘COLLECTOR’. Click ‘START’ once onto the next screen and wait until the temperature is green.

3. Once the sample is completely frozen, slightly open cap and put in a vacuum flask (shown below). These are near the lyophilizer. A Kimwipe and rubber band can also be used to make sure it is open (shown below). This is done so the vacuum can get in the flask and sublime the solvent. Up to 4 50 mL centrifuge tubes can be used in a vacuum flask at a time.



4. Attach vacuum flask to the instrument. This will be a tight fit but it needs to be to keep under vacuum. Make sure the flask is not open to vacuum by closing the knob.



5. Turn on vacuum by clicking on the red ‘HIGH’ under ‘VACUUM’ on the lyophilizer screen and set to 0 torr and press ‘START’. This can be done when the temperature is below -80 ºC (not what is on the screen below, just trying to take pictures for the SOP :)).

6. After setting to 0 torr, turn on the vacuum pump by pressing the green button with a light next to it on the pump. You will hear the vacuum start.

7. Open the vacuum flask to vacuum by turning the white knob on top of the instrumen. You can make sure it is pulling vacuum by slightly opening another vacuum line and touching it with your finger. After 15 minutes of the vacuum being on, come back and make sure the pressure is under 100 mTorr.

8. Come back the next day to check to make sure the sample is dry. If it is dry, turn off vacuum, vent and turn down the chiller. Dry the basin and, again, make sure the vacuum and chiller are turned off. It is very common to run it multiple times to completely dry the sample.

Common Problems

Problem: The solvent warms up too much during the drying process and turns into a liquid

Possible solution: Add less liquid to each centrifuge tube so there is less that needs to sublime. If this doesn't work, the schlenk line may be used in a similar fashion but having the sample submerged in liquid nitrogen while under high vacuum.

Notes:

Instrument obtained in N/A.
Manual written by Henry Thurber, June 2023.