Pros and Cons of Shell Freezing vs. Slant Freezing

Pre-freezing your samples prior to lyophilization is a very important step.

If the sample is not completely frozen, it can result in melt back during the process, damage the pump and produce a less-than-desired outcome.

There are three ways to pre-freeze samples: stub, slant and shell. Pre-freezing in a stub format, with the flask upright, results in reduced surface area and potential glassware breakage due to the expansion of ice during the phase change.

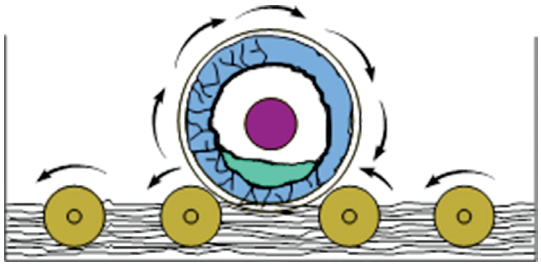
Because we do not recommend stub freezing, it is not included in this experiment.

Pre-Freeze Methods:

- **Stub freezing**
- **Slant freezing**
- **Shell freezing**

Stub

Slant



Shell

Slant or shell freezing the sample gives more surface area for sublimation and has several benefits.

1. This larger surface area increases the rate of sublimation which increases the evaporative cooling on the sample. The increased cooling lowers the sample temperature and helps prevent sample melt back, keeping difficult samples frozen throughout the entire freeze-drying process.

2. The increased rate of sublimation from the increased surface area results in a faster process and shorter run times.

3. Slant and shell freezing diffuses the ice expansion that occurs during freezing across a larger surface, preventing the flask from breaking.

Slant Freezing

To slant freeze, you simply position the flask with the liquid sample at an angle in the freezer. Position the glassware in the freezer at a tilt or use a Slant Flask Holder to provide a controlled and stable platform. A slant freeze flask holder is very easy to use and can be set to multiple angles. This protects the sample from spilling. The simple design provides a wide range of angles so the slant of each sample can be maximized.

More slant = more surface area = faster freeze drying

Shell Freezing

Shell freezing requires rotating a flask in a freezer or dry ice. The rotation can be manual or mechanical. A mechanical shell freezer takes the manual labor out of the process. The sample pre-freezes, typically to -40° C, on rollers to get an even shell frozen sample. This allows for maximum surface area and a thinner layer of sample for quicker freeze drying.

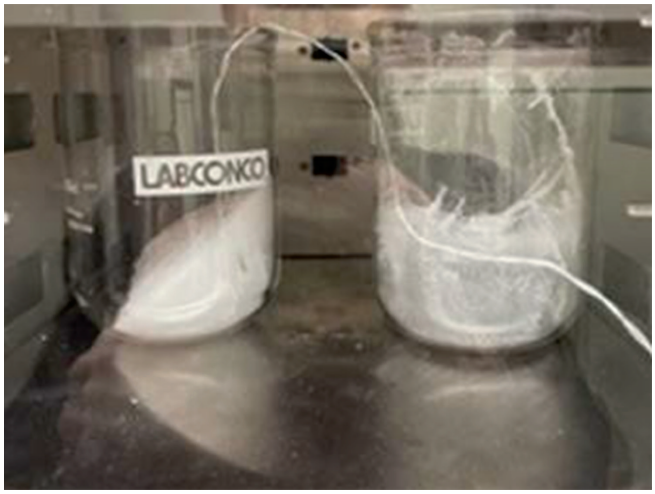
Shell Freezing vs. Slant Freezing

Goal: To determine the effect on time of shell pre-freezing versus slant pre-freezing.

Method: For this experiment, we placed two samples with 250 ml of water in each of two 600 ml flasks. One flask was frozen in a standard -80° C freezer on a slant. The other flask was frozen at -40° C in a shell freezer (7949020).

When both samples were completely frozen, and the freeze dryer was ready, the lyophilization process was started. The samples were placed in the 4.5L -105° C freeze dryer (720401000) with small tray dryer (780701000) and hybrid vacuum pump (7584000). The shelf temperature was set to +20° C and a vacuum pressure of 0.2 mBar. The heat allowed for heat transfer to the sample but was still at a low enough temperature and under a deep vacuum to prevent melting. After an hour, when no melting was observed, the shelf temperature was increased to +30° C.

At five hours, this picture was taken. Temperature probes were used to monitor actual sample temperature. At 5:00 pm, the slant frozen sample had a sample probe temperature of -32.2° C while the shell frozen sample had a temperature of 26.5° C. This discrepancy was due to the sample probe placement within the sample.



After 19 hours, the shell frozen sample volume was greatly reduced. The temperature of the slant frozen sample was -10.4° C and the shell frozen sample was 27.1° C.

The heat was increased to +50° C to help drive off additional moisture.

At 26 hours the shell frozen sample was completely dry and removed from the freeze dryer.

The heat was increased to +60° C and the slant frozen sample continued freeze drying. After 39 hours, the slant frozen sample was completely dry.

Conclusion: In this experiment, a shell frozen sample freeze dried 13 hours sooner, or 33% faster, than an identical slant frozen sample.

The greater surface area of the shell frozen sample versus the limited surface area of the slant frozen sample dramatically affects the required freeze-drying time. If you are looking for ways to speed up your freeze dry process, look at the way you pre-freeze your samples and you might find a time saver.