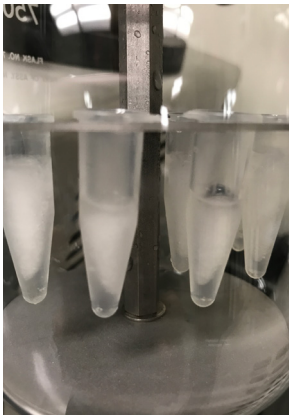
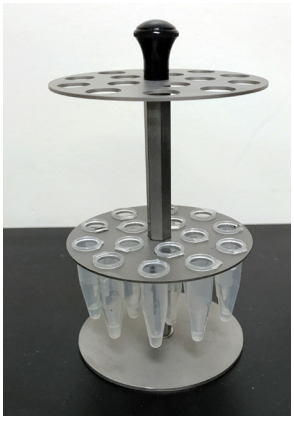
Lyophilizing Small Samples

Lyophilizing or freeze drying small samples in test tubes, micro centrifuge tubes or microplates can be difficult. The small volumes present two big challenges; keeping samples completely frozen during the start of the freeze drying process and keeping the dried samples in the tubes after they are dried.



Starting the lyophilization process with completely frozen samples is important and directly affects the overall quality of the finished product. Samples may begin to melt during the transfer from the freezer to the freeze dryer. The melting problem is more common when solvents are present, the sample is small or the sample is in plastic tubes. Once on the freeze dryer, the sample needs to remain frozen until a deep vacuum level is reached and lyophilization begins. If a melted sample is placed on the freeze dryer, you will notice bubbling liquid around the sample. If a sample has melted back, it can be more difficult to keep it within the tube as it will have the tendency to pop out of the tube.



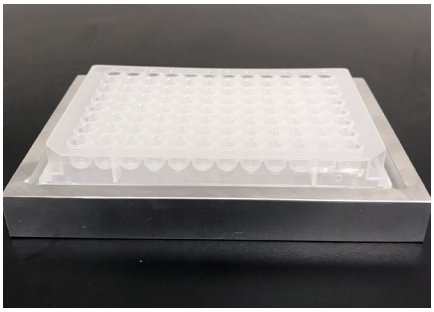
One way to maintain the sample temperature of samples in test tubes or microcentifuge tubes is to pre-freeze the samples insulated in ice. To do this, place your samples in a test tube holder.

Fill the flask with water about 1” at the bottom of the flask. Lower the test tube holder into the flask making sure the water does not get into the tubes when placed on a slant.

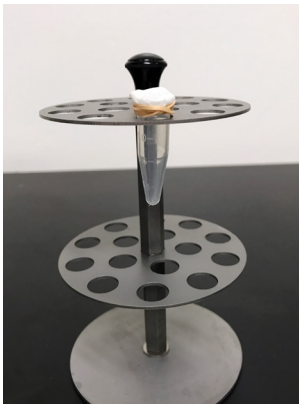
Place the flask without the lid in a freezer on a slant to prevent breaking. When the samples and water are 100% frozen, they are ready to be freeze dried.

The ice will keep the samples frozen during the lyophilization process.

If you are freeze drying microplates, it's especially important that the samples remain frozen during the process. By using a Microplate Holder, the plate and the holder are prefrozen together. The Aluminum mass of the Microplate Holder provides temperature stability to samples in microplates and prevents frozen samples from melting.



Sealing sample tubes with vapor permeable material improves recovery rates and eliminates the issue of samples leaving the tubes. Common materials used are Kimwipes® and Parafilm® poked with small holes.



LABCONCO CORPORATION
8811 Prospect Avenue
Kansas City, MO 64132
(800) 821-5525 | (816) 333-8811
labconco.com

20220429