

Freeze drying

Freeze drying is the first step in preparation for many analytical procedures. In contrast to oven- or air-dried samples, which are typically compressed and brick-like, freeze-dried samples are friable and sedimentary components emerge intact.

Procedure

Sample preparation (day before)

1. Samples must be fully frozen (preferably overnight), ideally on one or more of the stainless steel freeze dryer trays (so that the tray is also cold and samples don't melt as easily on the way to the freeze dryer).
2. Samples must be open to the air (for vapor to escape) but mostly covered (to prevent cross-contamination and particulates entering the pump). You can accomplish this by either poking a hole in the sample container (we have dental tools for the purpose) or removing the lid and covering the container with a lab wipe or paper towel, rubber-banded on.

Setting up freeze dryer (15 minutes)

1. If chamber is assembled but freeze dryer is turned off, remove transparent cover of chamber and set it upside-down on the bench next to the freeze-dryer. If necessary, lift out the metal tray frame and set it aside. Lift the chamber off its base and set it on the lid (to keep the greased gaskets from getting crud on them).
2. Remove metal elbow pipe from freezer chamber outlet and plug the hole with a rubber stopper.
3. Make sure drain cock is closed.
4. Open ballast (switch on front of pump).
5. Turn on vacuum pump and run for 15 minutes to warm up the pump and purge any water from the pump oil.
6. In the meantime, also turn on the freezer to cool down the condensation chamber. The temperature should be down to about -40° C before you start.
7. Turn off vacuum pump. Remove rubber stopper from outlet pipe and replace with metal elbow.
8. Move immediately to setting up samples.

Setting up manifold and samples (15 minutes)

1. Make sure the gaskets on the top and bottom of the chamber are free of dirt and particles and lightly coated with silicone vacuum grease. (Grease should only be applied occasionally.)
2. Place the manifold on the base, making sure that it is centered on the steel and not hanging over the edge.
3. Check that all ports on the manifold are set to the VENT position. (This sounds backward but "VENT" and "VAC" in this case refer to the vessels that can be attached to the ports.)

4. Put trays onto the tray rack and lower the rack into the freeze dryer (this is easier than lowering the chamber over the tray rack). Work quickly and don't let the samples melt.
5. Turn on vacuum pump. Immediately observe the top gasket through the clear manifold cover. You should see the black line (where the gasket meets up with the cover) getting wider as the chamber is evacuated and the cover presses down on the chamber. If the line is not getting wider, you have a leak. Find it and fix it right away!
6. Let the pump run with the ballast open for 10-15 minutes.
7. Close the ballast.
8. Run the freeze dryer overnight or until the vacuum gauge reads about 10^{-1} mbar.

Note: if the samples are very wet, or if there are a lot* of them, you may need to freeze dry them for several days. It is much more effective to defrost the chamber (see below) once daily rather than leaving the dryer to run on for multiple days without defrosting. The efficiency of water removal decreases once the inside of the condenser chamber becomes coated with ice (after 12-24 hours).

* "A lot" is more than about 250 mL of water, for example, more than 24 centrifuge tubes with 10mL of water, 2-3 trays of Poly-Cons with 2cc samples, or 12 specimen cups ¼ full of wet mud.

Shutting down freeze dryer (20 minutes)

1. Turn off vacuum pump and freezer.
2. Release vacuum slowly by turning one of the red caps on the manifold ports about ¼ turn until you hear it hiss. If you release the vacuum too fast, things will fly around in there.
3. Remove the cover and set it upside down next to the dryer. Lift out the tray rack and set it aside. Lift off the manifold chamber and set it on its upside-down lid.
4. Remove elbow pipe and replace with rubber stopper.
5. Open ballast.
6. Turn pump on and run for 15 minutes.
7. In the meantime, defrost the condenser chamber. Fill the condenser chamber about halfway with hot tap water. Allow the ice to melt, then dip out the water using a plastic beaker (the drain is completely plugged these days). Completely dry the inside of the condenser with a sponge and/or paper towels.
8. If you are putting samples back in, begin again at step 6 of "Setting up freeze dryer." You can put samples in as soon as the temperature gets to -40° F.