

Degassing Solvents – Freeze, Pump, Thaw

Degassing solvents involves iteratively applying vacuum to a liquid in order to remove dissolved gases such as O₂ or N₂ from solution. In order to prevent the liquid from also boiling away, the liquid is cooled to a temperature such that it has a very low vapor pressure, even under vacuum.

A common misconception of FPT technique is that you must *freeze* your liquid. **This is untrue**—you just need to **cool your solution** such that the vapor pressure of the liquid is extremely low under vacuum. Unnecessary application of extreme cryogenics to fully freeze some liquids is dangerous and is a common source of explosion/implosion accidents while FPT'ing. Don't do this.

A. General Safety Guidelines & PPE:

- Working with Schlenk and vacuum lines presents implosion/explosion dangers.
- Working with cryogenics presents cold burn and liquid oxygen dangers.
- Working with solvent pots involves the use of potentially pyrophoric material.

Full PPE (goggles or facemask, gloves, and labcoat) must be worn at all times, and manipulations should be carried out in a working fume hood. Work with the safety sash in front of you at all times. Be aware of the location and use of fire safety equipment in the lab prior to using flammables.

B. Instructions:

1. The liquid to be degassed needs to be in a vessel capable of withstanding prolonged vacuum: a solvent bomb, Strauss flask, Schlenk flask fitted with a greased glass stopper, or a round-bottomed flask with a 180 degree adapter.
2. Turn on your vacuum line (see Schlenk line and High Vacuum Line SOPs). Make sure that the traps on your vacuum line (either Schlenk or High vacuum line) are filled with LN₂ to prevent solvent from entering your vacuum pump.
3. Attach your vessel to the vacuum line.
4. Use a chemical bath (usually dry ice/acetone) to cool your vessel.

There are many options here, depending on your solvent. Keep in mind that you do **not** need to freeze your solvent, just significantly cool it. In the Tonks group, **using LN₂ to cool your vessel is banned**. Cooling a closed system with LN₂ is a recipe for condensing liquid O₂, and that risk is not worth saving a couple mL of solvent. With a few exceptions for very low-boiling solvents, cooling to -78 °C will result in only minimal solvent loss upon exposure to vacuum.

5. After your vessel has equilibrated with the cold bath, open it to vacuum.

- If your liquid is frozen, or is a very high boiling liquid, wait until your vacuum pump has returned to its normal “not actively pumping gas” noise or the gauge on your line reads its normal vacuum pressure.
 - If your liquid is not frozen and is a very low boiling liquid (think Et₂O), apply vacuum for several minutes.
6. Close your vessel and let it slowly warm back to room temperature.
 - **DO NOT** apply heat to make this go faster, you risk cracking your flask and causing an implosion. Watch to see if your solution is bubbling as it thaws; this indicates further dissolved gas is degassing from solution.
 7. After your vessel has returned to room temperature, repeat steps 4-6 3 times, or until the thawing solution no longer has visible bubbles.
 8. After your solution has been degassed, close the vessel to the vacuum line, and shut down your vacuum line as described in its SOP.

By signing below, you indicate that you have read and understand the content of this document.

Name: _____

Date: _____