

Degassing Solvents via Freeze, Pump, Thaw (FPT)

Degassing solvents involves pulling a repetitive vacuum on a liquid to remove saturated gases such as O_2 from solution. To prevent the liquid from boiling, it is cooled to a temperature such that it has a low vapor pressure. A common misconception is that you must freeze the liquid. You only need to cool the solution such that the vapor pressure is low under vacuum.

A. General Safety Guidelines & PPE:

- Schlenk and vacuum lines presents implosion or explosion hazards.
- Liquid nitrogen presents the risks of cold burn and liquid oxygen condensation.

Full PPE (goggles, gloves, and labcoat) must be worn at all times. 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. Make sure that the cold finger is trapped to prevent solvent from entering the vacuum pump.
3. Attach the vessel to the vacuum line.
4. Use a chemical bath (dry ice/acetone) to cool your vessel.

There are many options here, depending on the solvent. Cooling a closed system with LN_2 can condense liquid O_2 . With a few exceptions for very low-boiling solvents, cooling to $-78\text{ }^\circ\text{C}$ will result in only minimal solvent loss upon exposure to vacuum. After the vessel has equilibrated with the cold bath, open it to vacuum. If the solvent is frozen, or is a very high boiling liquid, wait until the vacuum pump has returned to its normal “not actively pumping gas” noise or the gauge on the line reads its normal vacuum pressure. Close your vessel and let it slowly warm back to room temperature.

- **DO NOT** apply heat to make this go faster, you risk cracking the flask and causing an implosion. Watch to see if your solution is bubbling as it thaws; this indicates further dissolved gas is removing from solution.
5. After the vessel has returned to room temperature, repeat steps FPT 3 times, or until the thawing solution no longer has visible bubbles.
 6. After the solution has been degassed, close the vessel to the vacuum line, and shut down the vacuum line.