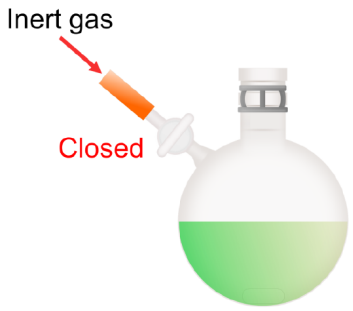
8: Freeze-Pump-Thaw

The freeze-pump-thaw (FPT) method is an effective way of degassing solvents, solutions, or liquid reagents.

Step 1

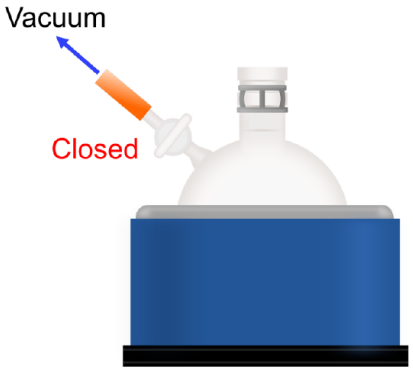
Close the stopcock on the Schlenk flask to seal the contents under inert gas. It is essential that the Schlenk flask is not open to inert gas whilst being frozen in liquid nitrogen (see Schlenk Line Safety for more information).



Sealing the Schlenk flask under inert gas.

Step 2 – Freeze

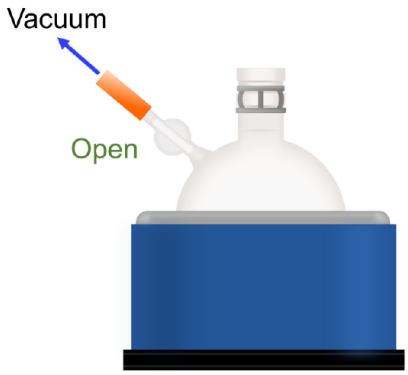
Cool the contents of the flask in a -78 °C dry ice/acetone bath. Cooling the solvent to low temperatures prevents or minimises the amount of solvent that evaporates when placed under dynamic vacuum. Liquid nitrogen can be used to completely freeze the contents of the flask, but this can lead to condensation of liquid oxygen if there is a leak present whilst the flask is under static vacuum. Liquid nitrogen should therefore only be used for more valuable liquid reagents or deuterated solvents.



Cooling or freezing the contents of the flask.

Step 3 – Pump

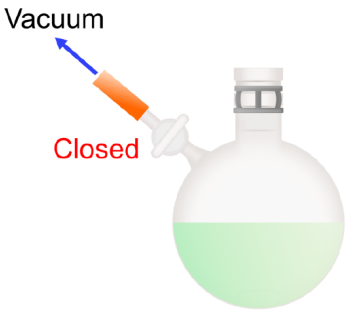
Once the contents of the Schlenk flask have cooled down or frozen, open the stopcock to evacuate the headspace. Keep under vacuum for 3-5 minutes whilst in the cooling bath.



Evacuating the headspace under dynamic vacuum.

Step 4 – Thaw

Close the stopcock to seal the flask under static vacuum and remove from the cooling baths. As the liquid slowly warms or thaws, any dissolved gas will escape into the headspace of the flask. Avoid applying external heat (warm water bath or heat gun) to make this process faster, since this risks cracking the flask and causing an implosion.



Thawing the contents of the flask.

Step 5

This process is repeated two more times for a total of three freeze-pump-thaw cycles – this ensures that all of the dissolved gas or any gas trapped within the frozen solid is removed. Once warmed back to room temperature after the final cycle, the flask can be backfilled with inert gas, or even a reactive gas such as H_2, CO or CO_2. It is essential to never backfill a flask with inert gas (nitrogen or argon) whilst frozen in liquid nitrogen since this can lead to condensation of the inert gas, which, if the flask is then sealed, may result in an explosion as the temperature increases.

Other Degassing Methods

Other methods of degassing solvents include: (i) sparging with inert gas; and (ii) the boil-degas method. For sparging, inert gas is simply bubbled through the solvent for an appropriate amount of time to displace dissolved air and oxygen. For the boil-degas method, the solvent is placed under dynamic vacuum for an appropriate amount of time (with a suitable solvent trap) to remove dissolved gases. Both methods can be equally as effective as the freeze-pump-thaw method but do result in significant evaporative loss of the solvent, and therefore is only recommended for bulk organic solvents and should not be employed for degassing expensive deuterated solvents or volatile liquid reagents.
