

Solvent Degassing by Freeze-Thaw

There are three fundamental techniques to degas solvent: inert gas bubbling in the reaction mixture (the best is Argon), ultrasound shaking under an Argon balloon and freeze-thaw technique. The first two are qualitatively less good, but can be used for less sensitive reactions, especially on large scale.

The following description is for the **Freeze-Thaw** technique:

Required for the technique:

- A Schlenk or a 2-Neck flask equipped with a closing neck, **evacuating through a septum is not good enough!**
- A "perfect seal" for the second entry of the flask. Either a glass stopper tightened with Teflon or grease or a new septum. A sealed tube with a second neck is probably the best (it is what the inorganic people use). **An already used septum is not good enough.**

Step 1: Close the flask. Freeze the solvent in liquid nitrogen



Step 2

Put the flask under vacuo (<0.5 mbar).



Step 3

Close the flask, warm up the solvent until everything is liquid again (obviously keep the flask closed, you are still under vacuo!). You can use a water bath to accelerate this process. To promote a better gas exchange, you can shake it a bit and you should not fill the flask more than 50% (the picture is therefore not ideal...)



Repeat steps 1-3 three times

At the end of the last cycle, freeze the solvent again (else it will jump in your line...) and put it back under inert gas before warming up. Once the solvent is liquid again, you are ready to start.