Solvent removal using a secondary trap

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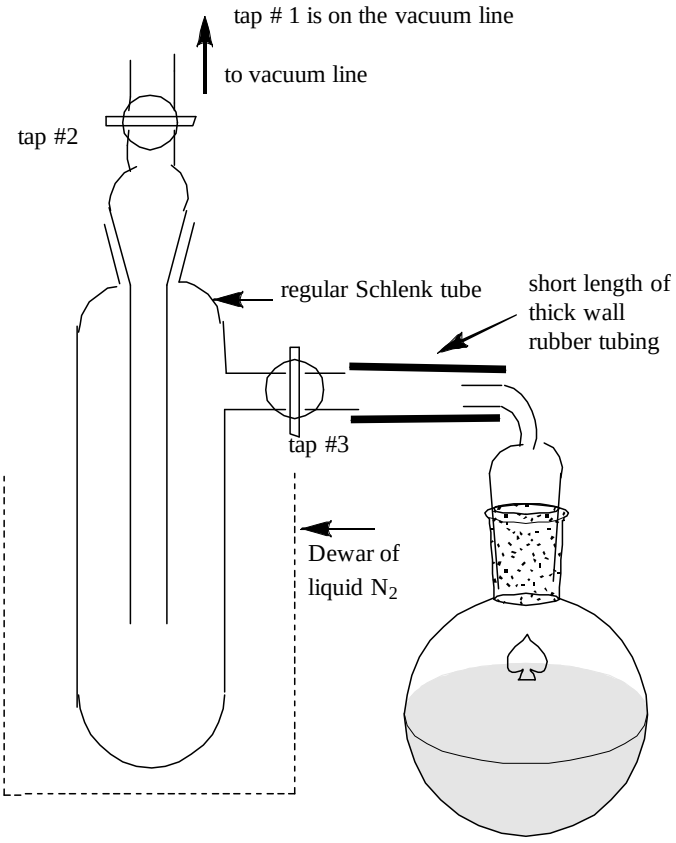
The usual method for removing solvent or other unwanted volatiles is to either use a rotary evaporator or a vacuum line. The choice between the two can often be influenced by volatility and toxicity. Vac lines are often placed in a fume hood whereas rotovaps may not be, so vacuum lines may be the better choice for toxic mixtures of solvents. Vacuum lines are usually capable of pulling a stronger vacuum so less volatile solvents will more easily be removed.

The problem with using a vacuum line for large volumes of solvents is that the trap that protects the mechanical pump will quickly become blocked with frozen solvent. To remove this entails a complete shutdown of the vacuum system. This is when a secondary trap can be employed to specifically catch the larger volume of solvent. It is also easier to dismantle, thaw and reattach if it does become blocked.

Another use for the secondary trap is for pulling off less volatile solvents. It acts as a short-path distillation in that the solvent vapour only has a little way to go before it is condensed in the receiving trap. This is much more efficient than trying to get that solvent all the way through the tubes of the vacuum line.



The diagram is probably fairly self-explanatory. The critical components are a regular Schlenk tube and a tube with a male joint that fits that of the Schlenk. Having a tap on that inner tube is useful but not mandatory. Try to choose the length of inner tube so that there is a reasonable gap between the end of the tube and the bottom of the Schlenk. The idea being that we want the condensing solvent to be exposed to a reasonable surface area cooled by liquid nitrogen before it has a chance to escape up the inner tube to the vacuum line. On the other hand, we need to be able to collect a useful volume of cooled solvent in the bottom of the Schlenk tube without blocking the inner tube.

As with all times when liquid nitrogen is used, remember that cooled glassware that is open to the atmosphere will condense liquid oxygen - possibly with disastrous results. Make sure that all cooled systems are under vacuum already and that the joint is greased sufficiently. In other words, as you begin the process of removing solvent, open tap #1 first and then tap #2. Once the secondary trap has been evacuated, place the liquid nitrogen dewar around it and then open tap #3. Reverse the order to close down the system.

This process can be successfully accomplished with air-sensitive materials too. Just let the system down to nitrogen (or argon) before closing the taps. If you are using argon (not available in the teaching labs), be very careful as argon readily condenses at liquid nitrogen temperatures.