

Standard Operating Procedure

Task: Setting Up Traps

Date: May 23, 2014

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Background:

- The use of traps on vacuum lines is essential to keep solvents from passing through pumps. Solvents can cause a dramatic decrease in the performance of pumps and in extreme cases can cause irreparable damage.

Training Requirements:

- Lab safety training

Potential Hazards:

- Liquid nitrogen burns
- Glassware under vacuum: implosion hazard
- Potential for oxygen condensation: explosion hazard
- Oxygen displacement

Special PPE Requirements:

- **Do not use gloves that will trap or absorb liquid nitrogen (cotton glovebox gloves, etc.)**

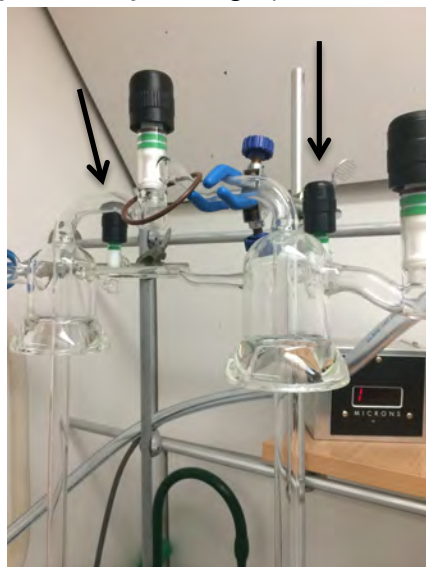
Materials Needed:

- Two vacuum traps, two rubber O-rings, two metal clamps, two vacuum-jacketed dewars, and liquid nitrogen.

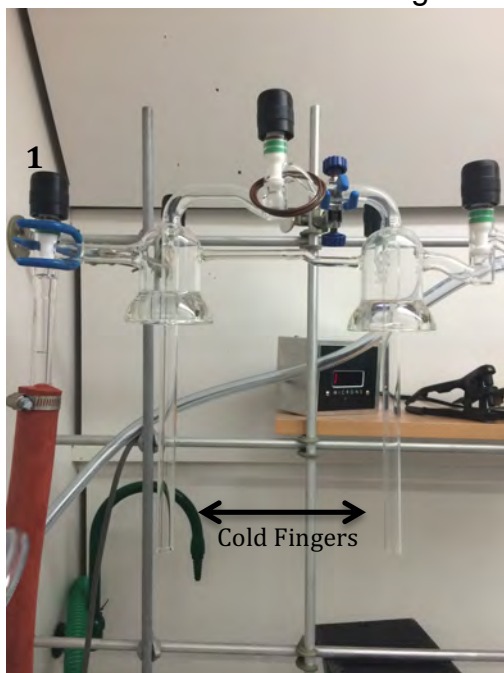


Procedure:

- Ensure that all stopcocks are closed before beginning, specifically the ones behind the traps (they are easy to forget).



- Take the large trap with the bulb at the base and put the rubber O-ring in the groove on top of the trap. Slide the trap up the cold finger on the left side, ensuring that the rubber O-ring is between the trap head and base. Secure with the metal clamp, screwing it tight. Support the base of the trap with your hand and make sure that the O-ring forms a good seal between the two glass joints.
- Repeat with the right (smaller, no bulb) trap, using the brown rubber ring to ensure a seal between the trap head and base. Secure with the clamp and support the base of the trap with your hand and make sure that the O-ring forms a good seal.



- Ensure that the Teflon stopcock connecting the two sides is closed.
- Turn on the vacuum pump (switch located below the hood sash on the left side). **Note: learn to recognize the typical operating sounds of your vacuum pump!** Aural cues are often the first sign that something is wrong. Every pump sounds different, so get to know the typical sound and be alert for changes. If “gurgling” sounds are persistent, this can sometimes indicate a leak. If “grinding” noises are persistent, this could indicate a problem with the pump.
- Open the stopcock labeled **1** in the picture above. The left trap is now evacuated.
- Evacuate the right trap by opening the stopcock connecting the left and right traps. Both traps are now evacuated. Your apparatus should appear as below.



- **Make sure the crossover valve and all of the ports on your line are closed.**
- Evacuate the line by opening the stopcock connecting the right trap to the line.
- Make sure that the Teflon stopcock leading to the digital vacuum gauge is open.
- Check the gauge: is the line pumping down properly? Gauges can shift over time, but typically the read < 50 mtorr (microns Hg). If the line is not pumping down fully, a leak is indicated: **find and resolve any leaks before cooling the traps.**
- Once the line is properly pumped down, fill the liquid nitrogen dewars approximately 1/3 full with liquid nitrogen.
- Carefully raise the dewars into place using lab jacks or platforms. **It is very important that both traps be evacuated before the dewars are raised to avoid production of liquid oxygen. If it is suspected or known that liquid oxygen has been formed alert researchers in the area and contact PI immediately.**
- Wrap towels around the tops of the traps to insulate.
- The vacuum should typically improve once the traps are cooled. The gauge should reflect this.



Dropping the Traps:

- Make sure that all potential users are done with the traps.
- Make sure that all ports are closed and that no flasks are currently being evacuated.
- Start with the trap further from the vacuum (right trap in the photo). Close the Teflon stopcock between the trap and the line (to the right of the trap in the photo) and close the stopcock between the two traps. **One trap is now under static vacuum: you must continue to drop the trap at this point, move quickly but carefully.** Remove the towel and slowly lower the dewar. Inspect the trap: the trap should only contain frozen solids — **if any liquids are observed (especially if a blue color is noted) immediately return the trap to its original condition (raise dewar and re-open to vacuum) and seek assistance for dealing with liquid O₂.** If only solids are observed, continue removing the dewar. Once the dewar is removed, open the vent stopcock (located at the back of the trap cap). A hissing sound should be heard as air is admitted into the trap. Unclamp the trap and carefully remove the trap and allow to thaw. Always thaw your traps inside of the hood.
- While dropping the right trap, the left trap was still under active vacuum (stopcock between the trap and vacuum was OPEN). This prevents any potential O₂ condensation in the second trap.
- Drop the second trap. Close the Teflon stopcock leading to the vacuum. The right trap is now under static vacuum: you must continue to drop the trap at this point, work quickly but carefully. Drop the trap following the same procedure as above: lower the dewar, check for liquids, vent and disconnect the traps.
- Once the traps are dropped, turn off the vacuum pump.
- Re-open the leftmost Teflon stopcock to allow air into the pump. **If this stopcock is not re-opened pump oil can be sucked up into the tubing!**
- Store O-rings off of the floor of the hood, preferably hanging loosely on the traps or on the clamps (clamped to the monkey bars).
Do not leave O-rings on traps while solvent is thawing. Do not let solvent thaw while traps are still connected to the line. These conditions can lead to over-pressurization and/or swelling of the O-rings.

Related SOPs and References:

- Liquid O₂ SOP
- Vac Transfer SOP