# Standard Operating Procedure

**Task:** Vacuum Transfer
**Date:** 5/26/2014
**Revision Date:** 05/06/2016

**Background:**

- A vacuum transfer (a.k.a. “vac transfer”) is essentially a low-temperature distillation.
- The “sending flask” contains a volatile solvent that is to be transferred to the “receiving flask”, which can be empty or contain a solid. Both flaks are placed under static vacuum and connected to each other in a closed system. The system is perturbed from equilibrium by cooling the receiving flask: the solvent vapors condense in the cold flask.
- Vac transfers are useful for moving volatile solvents between flasks without exposure to air, nitrogen, or any other solvent vapors (like those found in the glovebox). For example, vac transfers are commonly used to separate a solvent from the desiccant used to dry the solvent (i.e., separation of acetonitrile from molecular sieves). Vac transfers are also commonly used to prepare NMR samples in the absence of nitrogen, wherein a solid sample is loaded into a tube in the glovebox, evacuated, and solvent is added by vacuum transfer. The resulting sample is under vacuum rather than air, nitrogen, or argon.
- Vac transfers are performed using only glass-to-glass connections: never use rubber or plastic tubing to make a connection.
- The rate of transfer is limited by the size of the smallest opening in the path from sending to receiving flask. To avoid small channels in vacuum lines and simplify setup, **a U-tube connector is highly recommended**. U-tubes isolate the vac transfer from the rest of line, providing a short, wide path length for efficient transfers (as well as protection from condensing solvents or bumping solids into the main line). A U-tube is pictured below.

Image from Adams & Chittenden Scientific Glass: http://adamschittenden.com/Stopcocks%20and%20valves.html

**Training Requirements:**

- Lab safety training
- Work with a researcher experienced in the technique before working alone.

**Potential Hazards:**

- Implosion due to glass under high vacuum.
- Explosion due to liquid oxygen condensation.
- Cold burns due to cryogenic conditions.

**Special PPE Requirements:**

- **Face and/or blast shields if necessary**

**Materials Needed:**

- High vacuum line.
  Two flasks, one of which contains a volatile liquid.
- A cooling wand consisting of a spatula wrapped at one end with a pipe cleaner can be useful for spot cooling using liquid nitrogen

**Procedure:**

- Set up your high vacuum line and make sure that it is pumping down properly and that there are no leaks in the system.
- Assemble your vac transfer apparatus:
  - This procedure assumes that you will be using a U-tube, which is strongly recommended.
  - Clean, empty glassware should be oven-dried for at least 1 hour before use.
  - Attach the U-tube to the desired port on your line. Secure with a Keck clip.
  - Attach the receiving flask to the one side of the U-tube using an appropriate grease. The receiving flask should be a pressure flask equipped with a Teflon valve (such as a “bomb”, a Straus flask, or a J-Young tube). Secure with a Keck clip.
  - Attach the sending flask (containing solvent) to the other side of the U-tube. The sending flask can be a pressure flask equipped with a Teflon valve (such as a “bomb”, a Straus flask, or a J-Young tube) or a round-bottom flask equipped with a 180º Teflon-valve. Secure with a Keck clip.
  - **Note: the final transfer assembly is tall and heavy**! Use additional stabilization (such as rubber bands or a jack) if needed. It is also a good idea to clamp your U-tube if possible. Be sure that your apparatus will fit with your line before you start, including any jacks and cooling baths.
  - Once secure, evacuate the entire assembly up to the sending flask (**do not open the sending flask stopcock!**). Hopefully the glassware is still warm from the oven. If not, gently heat using the heat gun. Don’t rush this step; let everything cool completely and check your vacuum carefully for any leaks or other potential problems.
- Prepare the sending flask:
  - The sending flask should be under static vacuum. If the flask is under air or nitrogen, it must be degassed. Degas the contents using either the boil-degas method (not recommended for precious reagents or solvents) or the freeze-pump-thaw method.

    - Once degassed, close the Teflon valve on the sending flask and once again allow the full apparatus to pump down under active vacuum.
- Perform the vacuum transfer:
    - Place the apparatus under **static vacuum**. Isolate the system in such a way as to minimize total volume: if you have a choice, close the Teflon valve on the top of the U-tube rather than closing the valve on leading to the main manifold. Note that this process only involves closing a single valve: keep the valve leading to the receiving flask open, for example (and keep the valve leading to the sending flask closed).
    - Apply a dry ice / acetone cooling bath to the receiving flask. **Liquid nitrogen is not recommended because you are cooling a closed system under static vacuum**.
    - Apply a stir plate to the sending flask, if it contains a stir bar. This will enhance transfer rates and minimizing bumping.
    - Once the receiving flask is cold, **slowly open the valve on the sending flask.** This process is just like opening a flask to dynamic vacuum: opening slowly avoids bumping. Bumping is particularly painful in vacuum transfers, as it can push solids all the way across to the receiving flask — you will have to start over! So, slow and steady. (**Note: a glass wool plug can be loosely fitted in the apparatus if bumping is a big problem, to help knock down any solids that are strewn about during a bump.**)
    - Monitor the transfer. If the solvent is particularly volatile and your vacuum is good, your dry ice / acetone bath should start bubbling pretty soon after you open the flask. You will also likely notice that the solvent in the sending flask is starting to boil / bubble just like it does when solvents are removed under dynamic vacuum. These are good indicators that the solvent from the sending flask is being taken into the gas phase and condensed in the receiving flask. You can also lower the cold bath occasionally to look for condensed solvent.
    - Help the transfer. A spatula wrapped with a pipe cleaner is a great tool for vac transfers. You can dip the spatula in the cold bath, then rub the receiving flask above the level of the bath, cooling more of the flask. The cold spot left behind will often bead with solvent condensation on the inside of the glass. As the transfer proceeds, it is likely to slow down somewhat. Open the Teflon valve on the sending flask more as you go along. Do not adjust other valves or jostle the apparatus more than needed, as this can lead to leaks.
    - If the transfer slows down too much, there may be a leak. You can close the sending and receiving flasks and pump down the U-tube and try again. Or you can freeze-pump-thaw both sides and see if there is a deflection on the vac gauge (when frozen, just as when you started, there should be no gases present; if the gauge deflects, air has leaked into the system).
    - **Note**: when transferring solvents off of desiccants, do not transfer all of the solvent. This is especially true of ethers or other peroxide-forming solvents: any peroxides will concentrate as the transfer continues, and could detonate if taken to dryness.
    - Once you have transferred the desired amount of solvent, close the sending flask first. Allow a few minutes for any remaining vapors to transfer to the receiving flask. Then close the receiving flask and pump down the system. Depending on

your desired use, it may be useful to perform at least one freeze-pump-thaw cycle on the receiving flask (if no deflection is observed, there was no leak!).

- Cleanup:
    - Receiving flask procedure depends on your intended use. For the glovebox or future vacuum transfers, seal under static vacuum, remove from the line, and allow to warm until reaching room temperature. For Schlenk techniques such as cannula transfer, backfill with nitrogen before sealing and removal from the line.
    - Sending flask. For future vacuum transfers (if there is lots of solvent left), store sealed under static vacuum. If the flask is empty (or nearly empty), clean up or re-charge with solvent.
    - U-tube. Clean like any other glassware.

Another view of the full apparatus (using O-ring joints instead of greased ground glass):



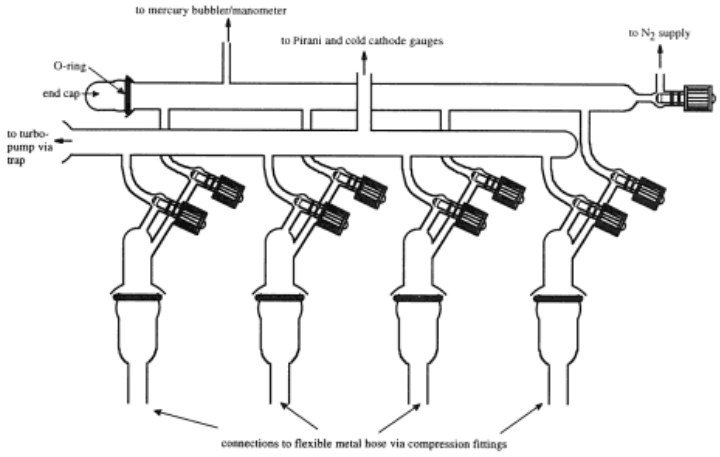


**Figure 5.20** Greaseless high-vacuum Schlenk line.

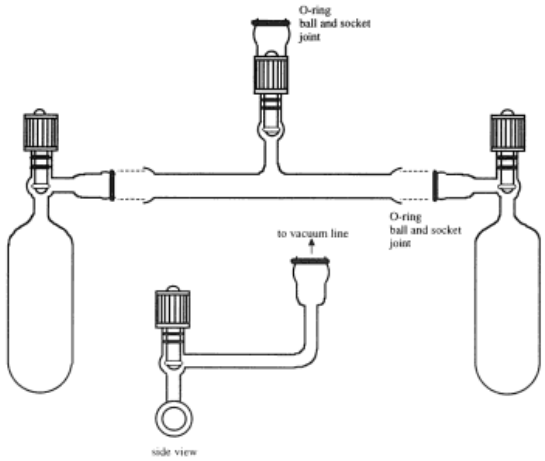


**Figure 5.21** Vacuum-transfer adapter for use on a greaseless high-vacuum Schlenk line.

From Advanced Practical Inorganic and Metalorganic Chemistry, by R. John Errington

**References and Related SOPs:**

- Setting Up Vacuum Traps SOP
- Degassing SOP