

## 8.5 - No-Air Techniques Guide

### Working on a vacuum manifold:

See LLP chapter 9.2 for an excellent discussion on working with air-sensitive reagents, including how to use a two-way manifold.

### De-gassing solvents:

The best way to remove water and oxygen from a solvent is to distill it over an appropriate drying agent (such as sodium). This can sometimes be a lengthy (and dangerous) task. Since we will only need a small amount of oxygen-free solvent in 5.301, it is more efficient to de-gas the solvent by purging with an inert gas, in this case N<sub>2</sub>.

- 1) Place some activated molecular sieves in a hot round-bottomed flask and purge with nitrogen until cooled to room temperature. Glass readily adsorbs moisture from the air, so it is important to thoroughly oven (or flame) dry all glassware. The sieves will act as a sponge to pick up water dissolved in the solvent.
- 2) When the flask has cooled, add your solvent and cap with a rubber septum. Secure the septum with copper wire.
- 3) Purge the solution by injecting a needle through the septum and placing it directly in the solvent. Vent the flask with another vent needle. You should see bubbles.
- 4) 15-20 minutes should be sufficient.

Another method of degassing a solution is "freeze-pump-thaw." The solution is frozen (over liquid nitrogen, for example), then vacuum is applied for several minutes. The vacuum line is closed, and the solvent is allowed to slowly warm to room temperature. Repeat the procedure at least two more times. *Caution: Some polar solvents like water, methanol, and acetonitrile expand with freezing and can break your glassware.* We won't be using this method in 5.301, but you may encounter it in the future.

### Cannula Transfer:

See LLP chapter 6.4

## Filtering a Solution:

Your filtrations apparatus will consist of three parts: the Schlenk flask containing the sample, the Schlenk frit, and the receiving Schlenk flask.

- 1) While the glass is still hot, grease both ends of the frit. Attach one end to receiving flask (don't forget the stir bar!) and cap the other end with another small flask that has a 14/20 opening. Secure with Keck clips. Evacuate the set-up. Refill with nitrogen (be careful not to suck oil from the bubbler into your manifold!). Repeat the vac-fill cycle at least three times.
- 2) When you are ready to filter, place both the sample flask and the receiving flask under a positive nitrogen flow. Quickly remove the capping flask from the frit and the septum from the sample flask. Connect the two components, and invert.
- 3) Close off the original sample flask from  $N_2$ , and pull a slight vacuum on the receiving flask (make sure you have a cold trap set up on your vacuum line, to prevent any solvent vapors from destroying the expensive pump!). You can then close off the vacuum, since filtration should occur under static vacuum.
- 4) When the solution has been transferred, re-apply a positive  $N_2$  flow and replace the frit with a rubber septum.

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