

Glovebox Training

4-28-09

1. When to use the glovebox
 - a. To weigh out reagents stored in the glovebox
 - b. To conduct reactions and/or workups with extremely air-sensitive compounds that cannot be handled using standard schlenk techniques
2. How to bring equipment into the glovebox
 - a. SIGN-IN
 - b. glassware/spatulas/solvent bulbs
 - i. Three 2-3 minute Evacuation and Refill cycles of the small antechamber
 - c. Solid reagents
 - i. Any solid should be rigorously dried under vacuum
 - ii. The solid should be placed in dried glass container covered with a rubber-banded kimwipe
 - iii. Three 2-3 minute Evacuation and Refill cycles of the small antechamber
 - d. Liquids
 - i. Solvent bulbs prepared according to section 5 can be brought in with three 2-3 minute evacuation and refill cycles of the small antechamber
 - ii. Unopened never-punctured sure seal bottles should be electrical taped and three VERY SLOW evacuation refill cycles should be done
 - iii. Any other liquids require consultation with the glovebox managers
 - e. General
 - i. Nothing should be brought into the box when the box has been exposed to solvent or while the box is purging
 - ii. Once you complete your cycles, the chamber should be backfilled with N₂ and turned to the off position
 - iii. Once you have brought your equipment into the box the door should be immediately sealed and the chamber should be left under positive pressure of N₂
3. Using reagents in the box
 - a. Solids
 - i. After weighing out any solids please return the chemical to its proper storage location
 - ii. Clean up the balance and glove box floor using the brush(es) and supplied masking tape
 - iii. Seal your flask
 - iv. If you have waste that is pyrophoric you may seal it in a provided plastic ziplock bag and then take it to your hood for proper disposal. Please consult with Melanie or the glovebox managers if you do not know how to properly dispose of such waste.

- b. Liquids
 - i. Before opening a liquid you must turn off the circulation in the box
 - ii. You may only dispense one bulk solvent at a time.
 - iii. Once you finish using one solvent, you must purge the box for the required time. You may not work in the box while purging.
 - iv. If you require using more than one solvent at a time you should
 - 1. Dispense solvent A from the bulk container to a personal vial and seal
 - 2. Purge
 - 3. Dispense Solvent B from the bulk container to a personal vial and seal
 - 4. Purge
 - 5. Repeat for each required solvent.
 - 6. Now you may use your personal supply of solvents at the same time.
 - 7. This process prevents cross-contamination of bulk solvents
- c. General
 - i. You should NOT bring in a solvent bulb to add solvent to a reaction that does not need to be run in the glovebox. These solvents can be added using standard schlenk techniques.
 - ii. You MUST NOT expose any bulk solvent to a solvent atmosphere!!
 - iii. Once you are finished in the box, any running reactions should be sealed with Teflon caps and wrapped with electrical tape or capped with an unpunctured septum before purging
- 4. Removing equipment/reactions/reagents from the box
 - a. The small antechamber should now be moved back to the off position
 - b. Anything you wish to take out should be placed in the chamber and sealed
 - c. Material should be IMMEDIATELY removed from the chamber and the chamber should be evacuated.
 - d. Sign out
 - e. Purge the box if you have used solvents and mark accordingly in the log book
 - i. Change the pressure levels to 12 –low and 13 high
 - ii. Open the vent at the top of the box
 - iii. Wait
 - iv. Close the vent
 - v. Return levels to 4 and 6
 - vi. Turn the circulation back on
 - f. Remember to stop your purge, ask your bench mate to remind you if you have a problem remembering
- 5. Storing chemicals in the glovebox
 - a. Solids
 - i. If you have personal solids that need to be stored in the glovebox you must contact the glovebox manager for a bin and ALL of your personal equipment and chemicals should be stored in that bin labeled appropriately

- ii. If your chemical must be kept in the freezer you will be assigned a space in the freezer
 - b. Liquids
 - i. Personal liquids should be stored in Teflon capped vials and secured with electrical tape. These vials will then be stored in a personal jar with a Teflon cap which can be acquired from the glovebox managers.
 - ii. Bulk, non-volatile, non-coordinating solvents may be stored in the glovebox in solvent bulbs with flat bottoms capable of standing upright.
- 6. General
 - a. No chemicals/reactions/vials/etc should ever be left on the glovebox floor when you are not actively working in the box
 - b. Clean up after yourself and remove all trash as soon as you are finished in the box
 - c. Stocking the box
 - i. Vials/pipets/caps/kimwipes/jars/bins/lids are all provided by the glovebox managers.
 - ii. DO NOT restock the box yourself
 - iii. If you notice something is getting low, feel free to remind the managers, they will be appreciative
 - d. If you need to use the large antechamber consult with the glovebox managers.
 - e. If you get a hole in the gloves, IMMEDIATELY notify the glovebox managers
 - f. When in doubt, ask for help!
 - g. Solvents that should never be used in the box
 - i. Methanol, acids, water
 - h. Chemicals that should only be used in the box in small quantities with rigorous care
 - i. Methyl triflate, methyl iodide