

Glovebox General Use

Glovebox:

Enter and exit the box slowly: The box is not perfect, there are small holes that let ambient atmosphere into the box. Therefore, the box should always be kept under a positive pressure, i.e. not ripping your arms out of the gloves and dropping the glove box pressure lower than atmospheric pressure. If this happens too quickly, and the box cannot compensate quickly enough, it is possible that ambient air could enter the box under the reduced pressure (through the small holes), therefore raising oxygen levels.

Antechamber:

Evacuate the Antechamber: The antechamber is to be kept under vacuum at all times (except for putting things in the antechamber or removing them from the antechamber). This is standard practice for most antechambers for the following reasons: (1) It requires the glovebox user to refill it prior to opening the door as a defense against moments of carelessness and (2) it prevents leakage of outside air into the glovebox. **Evacuating the antechamber should be the first thing you do after closing the antechamber door.** IT SHOULD BE A HABIT! It is orders of magnitude better to accidentally evacuate the antechamber and have to waste a little nitrogen refilling it than it is to forget to evacuate the antechamber. Even if you are coming back out of the box in 5 minutes, EVACUATE THE ANTECHAMBER!

Reduce the amount of time the antechamber is left open by having all items you wish to bring in the box ready.

Short Cycles are Inexcusable: As all of you should be aware, we use **three 10-minute cycles on the small antichamber** and **three 15-minute cycles on the large antechamber** to pump materials into the box, only refilling the chamber halfway (i.e. to the -10 psi mark on the manometer) between cycles. The large antechamber should first be filled with air. You will be opening the chamber to air to bring in your items so using Argon is simply wasteful.

There are several reasons for this protocol:

(1) It allows the antechamber to reach its minimum pressure (c.a. -30) on both the second and third cycles. The minimum pressure represents the minimum amount of oxygen possible to introduce to the glovebox. If the antechamber never hits the minimum pressure, you will bring oxygen into the box.

(2) Cycling the antechamber removes more water from the antechamber than a single cycle.

(3) It's a lazy way to purge a little bit of the box atmosphere.

The central point is this: **Never, ever go into the box using less than 5 minute cycles!!!** Even if you only want to bring stuff out of the glovebox. There are NO circumstances where short cycles are justified.

If you are bringing in Kim wipes or other porous materials (cork rings, etc) you must do a 15 min purge as normal, but then leave it in the antechamber for a minimum of 12 hours. Also make sure you have

dried the Kim wipes in the oven for 2 hours (making sure to remove the plastic on the box) before placing in the antechamber.

Proper Logbook Use: Anytime you use the antechamber, record it in the logbook. This is the best way to prevent atmosphere contamination. These must be accurate! You must record the date, time, item brought in (or out), whether you used a solvent (for the N₂ box), and what the O₂ and H₂O levels were before and after usage.

Check the logbook before pressurizing the antechamber. If someone else is using the antechamber, check with that person before using the antechamber. Failure to check the logbook will result in contaminating the glovebox atmosphere. The log book also provides record of usage making it easier to troubleshoot and correct any problems.

The logbook is there for a reason. Use it!

Glovebox Etiquette and Further Protocol:

Clean Up After Yourself

This is community equipment, not your benchtop. If you want to be a slob, be a slob on your own time, not other peoples. Use third grade rules when using the box: Return it better than when you found it. LABEL EVERYTHING OR IT GETS THROWN AWAY!

Pipet Tips are Brittle! They break extremely easily. Broken glass damages the gloves and contaminates materials.

Pipets lying around with bulbs still attached to them imply that you're planning on reusing the pipet. It implies that you are contaminating the solvents that require time and effort to get into the box in acceptable quality. There should always be fresh pipets in the box, so use as many as you need.

Loose vials both on the floor and on the shelf clutter an already limited work space for the next user. It also makes the glovebox difficult to clean. Please pick up all loose vials and put them in a tray. If your tray is full, remove unnecessary vials to make room or get a new tray. Loose vials will be thrown out when the box is cleaned.

If you use the last of the vials or pipets, replace it with one from the oven. Refill the empty can and place it in the oven to dry.

All waste should be immediately removed from the box! If you use a chemical or solvent and leave the waste lying around you are still contaminating the box atmosphere with whatever you used.

Carelessness Kills

Balances are sensitive instruments. When they are not level, the weights they read are not accurate. The floor of the glovebox is uneven. When you move the balance, it is likely to not be level. If you want the weights you record to have useful physical meaning, then you don't want this. DON'T MOVE THE

BALANCE! Keep the balance doors closed at all times!!! This helps to prevent miscellaneous dirt from contaminating the balance which, of course, affects the weights you record.

Open solvent bottles (or open reactions, including venting with a needle stuck in the septum) are especially bad in a glovebox because it is nominally a closed system. In other words, the atmosphere saturates in solvent vapors. This then leads to the catalyst bed becoming saturated in solvent vapors, which leads to shorter bed lifetimes. The current cost to rebuild a catalyst bed is A LOT! Also, solvent contamination becomes a problem (ether, for example, gets into everything easily). Therefore, the box should be purged after usage of solvents or volatile chemicals (not including after using amines, phosphines, etc. see later section on these).

Needles are sharp. They are the single largest hazard to the gloves and the survival of our world. If you want to use a septum with a needle through it to dry something under vacuum, please don't push the needle all the way into the septum. When you do this, the needle sticks out past the end of the septum, just begging for a glove puncture.

Make sure that the trap is iced up before using the pump. Make sure that the pump is on before opening any of the vacuum valves. Make sure that the trap is iced up while the pump is on. Don't leave the pump on if you aren't using it. If the trap isn't cold, the trap contents all end up in the pump oil. If the pump isn't on, using the vacuum system will result in contamination of the glove box atmosphere with outside air.

Label and cap your samples. Unlabeled samples disappear sometimes. Uncapped samples are almost certain to end up either in the trashcan or scattered all over the box.

Amines, phosphines, and titanium compounds are very problematic. Not only do they bind very well to the catalyst bed (amines and phosphines), but they leave a white film on everything in the box. When using these compounds (or any other volatile chemical), shut off the blower and close off the catalyst bed. After using these compounds, purge the box and reseal the bottles/vials tightly with black tape. Material that came into contact with these compounds should also be removed immediately. Everyone is responsible for their own trash.

Flasks being brought into the glove box are subject to high vacuum, and therefore need to be appropriately sealed to prevent them from busting open in the antechamber. When bringing a sealed flask into the glove box, ensure that it has been completely evacuated on your line. When bringing solvent into the glove box (i.e. THF), fill the bomb flask no more than 1/2 way and thoroughly degas then solvent before placing it in the antechamber.

Volatile Chemicals

You must turn off the blower and close the valves to the catalyst prior to using phosphines, amines, and chlorinated solvents. These are all detrimental to the catalyst. You must do this when using the above. If you have questions about a compound in mind, ask a senior member.

When done using the above chemicals, purge the box for atleast thirty minutes.

Vacuum and Solvent Removal

To remove solvents, you must use the vacuum. Assemble the cold trap and turn the vacuum on and screw the top vent closed. Clamp the trap to the top portion prior to submersion in liquid N₂. Allow vacuum to be established.

Submerge the cold trap in N₂ using a dewar.

Open the swage lock that goes to the box. Allow the vacuum to be reestablished before opening the swage lock inside of the box.

Do as you please with the vacuum. Be aware that the glass fittings should always remain on when the vacuum is NOT being used. This prevents any leak the brass fittings may have entering the box.

When done with the vacuum. Make sure all the swagelocks in the box are closed.

Reverse the first four steps. Make sure the N₂ is removed and open the vent. If the liquid N₂ is not removed, this could condense liquid oxygen which can be explosive, especially in the presence of any organic solvents you may have just put into the cold trap.

Change the pump oil every month as it is used quite heavily.

Patching Holes

If you poke a hole in the glove box and DON'T DO ANYTHING ABOUT IT, YOU WILL BE AT THE MERCY OF THE GROUP! TELL SOMEONE OR DO SOMETHING ABOUT IT IMMEDIATELY. INACTION IS FAR WORSE THAN CREATING THE HOLE ITSELF. Mistakes happen, but do something about it, no one will yell at you for trying to fix the problem, or getting help to alleviate it.

Notify the people responsible for the glovebox and in the mean time cover the patch to prevent the O₂ levels from going too high.

If the hole is much too large to patch, find a senior member quickly. If it is late at night, use the port hole that is found in the box and close off that port. Leave it there, go home, and tell someone in the morning.

Purging the Box

1. Turn the blower off
2. Close the hand valves to catalyst bed
3. Adjust minimum pressure to 3.5 and maximum pressure to 5.
4. Open purge valve 1/2 way
5. Allow box to purge for about 30 min or until oxygen level returns to normal

6. Close purge valve

7. Open hand valves on catalyst bed

8. Turn blower on

NEVER leave the glovebox purging unattended!