SOP for Glovebox Use

General Glovebox Information

You are not permitted to use the glovebox without first undergoing training by one of the people in charge of the glovebox.

The glovebox should be used to conduct reactions and/or workups on air-sensitive compounds that cannot be handled using standard schlenk technique. Care should be taken at all times to exclude water and O₂ from entering the glovebox.

Handling Solids in the Glovebox

1) **Preparing solid compounds for glovebox**: Before bringing any solid compound into the glovebox, it needs to be dried on a high vacuum line to remove any water or solvent. If it is a new bottle purchased from a chemical company, it can be brought in as is. After ensuring that there is no water/solvent, the O₂ in the container needs to be removed before being brought into the glovebox. To do this, remove the cap from the container and stretch a piece of Parafilm over the opening of the bottle. Next poke 5–10 holes in the Parafilm to allow for gas flow into and out of the bottle.

2) Sign into the glovebox logbook. The small chamber should be under constant vacuum when not in use, so turn the handle to refill and then to close before opening the door (You don’t want to leave the small chamber under refill when you open the outer door to air. This would create an open “hole” from the box to the air).

3) Next place the bottle with Parafilm and cap on the tray inside the small chamber. Replace the door and turn the handle to evacuate. Wait 3–4 minutes before refilling the chamber with N₂, then evacuate again. Repeat this evacuate/refill cycle 3 times.

4) After the final cycle, refill and turn the handle to close. You can now remove the inner door and bring your solid compound into the glovebox.

5) When you finish weighing out/handling any solids in the glovebox, make sure you have cleaned up any solids on the floor of the glovebox or the balance. If you have spilled a large quantity, use the brush to gather all solids into a pile and collect onto a piece of masking tape. For smaller spills, use the vacuum cleaner located in the glovebox. Return all vials to their appropriate bins. Before exiting the glovebox, take with you any Kimwipes, pipettes, or other trash. Return the small antechamber to evacuate and sign out of the glovebox in the logbook.
Current glovebox managers: Chelsea, Monica L., and Naoko

6) If you are storing any solids in the glovebox, make sure they are in your personal bin or the appropriate bulk bin and not left on the floor of the glovebox.

Handling Liquids in the Glovebox

1) All liquids that are brought into the glovebox must be dry and degassed (no dissolved O\textsubscript{2} in the liquid). If you need help with learning how to dry or degas a liquid or want to know if it is okay to bring a specific liquid into the glovebox, please ask one of the people in charge of the glovebox. You should not bring in a solvent bulb to add solvent to a reaction that does not need to be set up in the glovebox (use standard schlenk techniques when possible).

2) Protic solvents (alcohols, acids) may only be used in the single person glovebox.

3) Bringing in a Schlenk Flask: It is preferable that liquids be brought into the glovebox using a Schlenk tube/flask with a Teflon screw cap. These vessels have high vacuum seals that can be brought into the glovebox using the same protocol as is used under the handling solids section (evacuate/refill x3, 3–4 min/cycle). If you have not used this type of vessel before, again please ask one of the people in charge of the glovebox. When these vessels are sealed improperly the catalyst and pump can be damaged, so please ask for help if you need it!

4) Bringing in a bottle or vial: If you want to bring in an unpunctured sure seal bottle or you are bringing a vial containing a dry, degassed liquid under an inert atmosphere (with a Teflon lined cap! ie. new deuterated solvent bottle), use the short cycle system. This consists of slowly evacuating the antechamber to the halfway point before refilling. Repeat 10–15 times.

5) Storing/handling liquids in the glovebox: If you want to store a liquid in the glovebox, it must be in either a schlenk tube or a vial with a Teflon lined cap. If in a vial, the seal between the vial and the cap needs to be electrical taped and stored in a secondary container. Do not open multiple bulk solvent bulbs/vials at the same time. This can result in cross-contamination. If you need to use multiple bulk solvents, you must purge between opening each container (ie. Open solvent A, dispense into personal vial, purge, open solvent B, dispense into second personal vial, use both A and B simultaneously). If using your own vials, you may use multiple solvents at the same time.

6) Opening containers of liquids: Sign into the glovebox logbook. The small chamber should be under constant vacuum when not in use, so turn the handle to refill and then to close before opening the outer door. Place the vessel containing the liquid in the small antechamber and replace the outer door. Use steps 3 and 4 in this section to determine how long/how many times to evacuate the chamber. Once you have properly cycled in your vessel or if you’re using a liquid already in the glovebox, turn off the circulator before opening the vessel
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(don’t forget to do this!! this closes the opening to the catalyst bed). When you have finished, any running vial reactions should be sealed with a Teflon lined cap and wrapped with electrical tape. Place anything you wish to take out of the glovebox (including all used pipettes!) into the antechamber and replace the door. Material should then be removed and the antechamber placed under evacuate. Sign out of the glovebox logbook.

5) **Purging the glovebox:** The atmosphere is now contaminated with solvent and needs to be flushed out. To purge, first increase the pressure settings to 12–13 mbar. Next, open the lever at the top of the glovebox. Wait the specified time (varies per solvent—see table below). When this time has passed, first close the lever, return the pressure to 4-6 mbar, and turn the circulator back on. (For the single person glovebox, simply press the purge button to initiate the purge. There is no lever.)

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Type</th>
<th>Purge Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene, Pentane, Hexanes, Alkanes, Ether, THF, Acetone</td>
<td>Noncoordinating</td>
<td>10 min.</td>
</tr>
<tr>
<td>MeCN, chlorinated compounds, DMSO, amines</td>
<td>Coordinating</td>
<td>15 min</td>
</tr>
<tr>
<td>Phosphines, volatile sulfur compounds</td>
<td>Catalyst Poison</td>
<td>25 – 35 min.</td>
</tr>
</tbody>
</table>

Note: These are approximate purge times. Solvent purge times should be based on the volatility of a compound, the coordinating ability to Cu, and the length of time the liquid is open in the glovebox.

**Other Comments**

The sign up sheet should be used to schedule the time you plan to use the glovebox each day. Maximum sign up time per day is 3 h during prime time hours, 9–6p. If you finish with the time that you scheduled earlier than expected, be courteous and let the next person signed up know.

Do not purge both double person gloveboxes at the same time. There is not enough N\textsubscript{2} pressure provided to that room to maintain both glovebox purges.

Do not work in the glovebox while it’s purging.

The glovebox gloves should remain chemical free so don’t wear a labcoat in the glovebox unless it’s designated for glovebox use only.

Stocking the box: Vials, pipettes, caps, kimwipes, jars, and bins are provided by the people in charge of the glovebox. Please do not restock the glassware yourself. If something is getting low, let one of them know.
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If you need to use the large antechamber consult with the glovebox managers.

If you make/find a hole in the gloves, notify the glovebox managers.

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.

Finally, if you have an accident in the glovebox (spill solvent in the antechamber or in the box, leave a door to the antechamber open, etc.) **tell the glovebox managers immediately.** We won't be angry at you and it's better to immediately deal with the problem so that the catalyst and pump are not damaged.