

## Glovebox etiquette in the Lewis group

Introduction: The gloveboxes are here to assist you in oxygen and water sensitive chemistry. The following guidelines are given to protect the box atmosphere and longevity and ultimately your chemistry and that of the whole group. If you feel that these guidelines impede your ability to do work, talk about it with the person in charge of the glovebox and they will help assist you in designing a set up that will work for you! Disregarding the guidelines compromises your chemistry as well as everyone else's.

### Available boxes:

\*Big Bird – Vac, purchased 2008. Drybox, Ar. Noyes 227B. Use for electrochemistry.  $O_2 < 0.5$  ppm

\*Snuffleupagus – Vac, older than time. Drybox,  $N_2$ . Noyes 227B. Use for electrochemistry and storage. Absolutely no bad solvents/chemicals with the exception of  $CH_3CN$  open for  $<5$  minutes at a time.  $O_2 < 1$  ppm

\*Oscar the Grouch – Vac, older than time. Flushbox,  $N_2$ . Noyes 227B. Alcohol OK. Electrochemistry, set-up, surface chemistry.  $O_2 < 10$  ppm

\*Elmo – Vac, purchased 2008. Drybox,  $N_2$ . BI 032. Electrochemistry and chemical synthesis.  $O_2 < 0.5$  ppm

\*Count von Count – Mbraun, less old than time. Drybox,  $N_2$ . BI 032. Don, Tony, electrochemistry and set up.  $O_2 < 1$  ppm

\*Cookie Monster – Vac, eh? As old as time?. Flushbox,  $N_2$ . BI 023. Alcohol OK. Electrochemistry, set-up, surface chemistry. Connected to XPS.  $O_2 < 10$  ppm

Note: Contact Box Czar before using a new box – even if you are trained on the old box. If nothing else, so they can keep track of who is using what and include you on important info for that box.

### Guidelines:

- 1) Log book: Always check and fill out the logbook when bringing things into or out of the box, turning off circulation, and purging. The person in charge of the glovebox will also note things like maintenance and glovebox regeneration in the log book. If someone else is bringing things into the glovebox, you will have to wait until they are done to begin bringing your things in. If you notice strange glove box behavior (noises, spikes in the atmosphere, etc. note them in the log book)
- 2) Bringing things in and out of the box: you must do three pump down/backfill cycles before bringing anything into the glovebox, and the pressure must go down to  $<50$  mTorr before each backfill. If there is no manometer 15 min for general items in the large chamber and 5 minutes for the small. Porous items will take longer and sometimes overnight. Have the person in charge of the box show you how to do this before you attempt to use the box. It is important to observe recent antichamber activity before opening the chamber to the box – if someone recently

went out of the box (<15 min for lrg chamber, <5 min for small) you need to wait or do backfill cycles before opening the antichamber to the box.

- 3) Vacuums: NEVER use the antichamber vacuum to remove solvent
- 4) What goes in the box:
  - a. Glassware - DRY. Glassware should be dried in an oven for at least a few hours before bringing it into the box. If you cannot do this, you can remove much water from the glass by doing a rinse with EtOH or MeOH then hexanes. There is a ton of water in glass – you can easily see this by running a flame over a typical piece of glass.
  - b. Chemicals – nonvolatile solids should be opened and covered with a kimwipe/rubberband unless packaged under inert atmosphere. Volatile solids and liquids should be transferred to a shlenk flask, degassed, then brought in under inert atmosphere – again, unless the purchased chemical was packaged under inert atmosphere.
  - c. Porous things should be kept to a minimum and require overnight pump-down (i.e. gloves, boxes of kimwipes, etc.) Many groups go a far a to dry kim wipes in an oven before they bring them into the box. There is no reason to bring in waste tags in my opinion, chemicals become waste as soon as they leave the box and waste tags just introduce extra water/O<sub>2</sub>. Label your waste with what is in it and ‘used.’ Do not bring rolls of labeling tape or markers into the box – instead use a wax pencil (available at chemistry stockroom) Tape and markers cannot be evacuated thoroughly and sometimes markers explode and leave a huge mess. Marker ink also contains alcohols which are on the ‘what not to bring in’ list. The wax pencil needs to be pumped down overnight. Sieves should be dried overnight by heating in a Schlenk tube under vacuum. Bring them into the box under inert gas.
  - d. Large equipment (ring stands, lamps, hotplates, etc.) run it by the person in charge of the box. There is limited space in the box, and the person in charge may know if there is something similar already present or of a smaller volume alternative. If not, they’ll probably gladly have you bring it in!
- 5) Good solvent/Bad solvent
  - a. Traditional ‘good’ solvents: pet ether, diethyl ether, THF, benzene, toluene and solvents of the same family as those listed.
  - b. Traditional ‘bad’ solvents: methylene chloride, acetonitrile, alcohols, amines, water, and any other solvent that is oxidizing, coordinating, or otherwise damaging. These solvents reduce catalyst lifetime and it’s ability to keep the box atmosphere clean and they can react unfavorably with others’ chemistry. These solvents can be used after turning off circulation, and the box must be purged after the solvent has been closed and all traces are removed from the box (see blow under circulation and

purging). NO ALCOHOLS OR WATER is allowed in any Lewis group drybox under any condition – if you cannot deal with this you need our fearless leader's (Nate's) approval before using these items. These items can be used in the flushboxes. Bad solvents (with the exception of CH<sub>3</sub>CN are not allowed in Snuffleupagus, the old drybox)

- c. Acetonitrile: Acetonitrile is very important to our group and our chemistry. For this reason, the following rule apply – if the acetonitrile is open <5 minutes it can be used without closing circulation or purging. If >5 min <1h close circulation and purge when you are finished. If >1h, you must figure out a closed system to do your electrochemistry in.
- 6) Circulation and Purging: Preserving glovebox atmosphere is important to your chemistry, the group's chemistry, the ability for the catalyst to maintain an O<sub>2</sub>/H<sub>2</sub>O free atmosphere, and the longevity of the catalyst! Remember what is in the atmosphere is in your chemistry and everyone else's!
- a. There are instructions posted on every box with this capability.
  - b. Circulation: Circulation is controlled by a blower, make sure this is OFF when the inlet and outlet to the catalyst are closed, otherwise the blower will break and the box will be out of commission for a long time.
  - c. When to do this: The circulation should be turned off before opening any bad solvent or chemical – if you are unsure, ask – after every trace of that solvent is gone (all bottles and echem cells are closed, and all contaminated kim wipes, pipettes and secondary containers are removed from the box start the purge. General purge guidelines are as follows:
    - i. Single box: >15 min after volatile bad solvents/chemicals, >20 min after less volatile solvents/chemicals
    - ii. Double box: >30 min after volatile bad solvents/chemicals, >40 min after less volatile solvents/chemicals
  - d. When in doubt, purge longer – N<sub>2</sub> is much cheaper than a new catalyst
  - e. Do not turn circulation off for >1 h before purging. Atmosphere will be compromised in > 1 h.
  - f. Keep the fridge closed while bad solvents are open/the box in purging.
- 7) Trash and Cleanliness: Each user is in charge of removing their own garbage in a timely manner. Each user is also in charge of keeping common areas clear when they aren't working. This means store your things in a bin!
- 8) For more info on glovebox etiquette, please see the Bercaw group guidelines ☺

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Thank you for reading and happy chemistry!