Miller Lab Glovebox Manual

Table of Contents

I. Introduction

A. Principles of glovebox operation

This manual provides an overview of proper operating procedures for the Miller group gloveboxes. New users will be introduced to the best practices for operating and maintaining gloveboxes. Veteran users will find useful reminders and lab-specific details. So, what is a glovebox? A glovebox is an enclosed plexiglass-and-metal workstation that provides an oxygen-free environment (an “inert atmosphere”) by virtue of a catalyst bed (“purifier”) that utilizes activated molecular sieves and copper oxide to remove water and dioxygen from the inert gas (dinitrogen or argon). Materials can be brought in and out from the inert atmosphere using antechambers, which act like air locks to exclude oxygen before exposure the glovebox atmosphere. Each Miller group glovebox caters to slightly different users, but each glovebox is ready to be used to tackle a wide array of reaction chemistry. The gloveboxes are fully equipped “wet” boxes: each has a –30 ºC freezer, cold well, balance, auxiliary vacuum feedthrough, vials, pipets, kimwipes, stir plates, ring stands with clamps, celite, alumina, sieves, and a selection of solvents. This manual outlines glovebox operating procedures designed to provide a safe and efficient working environment for all users. ***All new users must read this manual in its entirety, and also undergo formal training from the appropriate group member in charge of each glovebox***. The core training only needs to be done once; shorter box-specific trainings must be completed for each additional glovebox to be used.

B. Quick Reference

Table 1. Guide to chemical compatibility with catalyst.

Functional Group	Examples	Purifier (Open/ Closed)	Notes
Saturated hydrocarbons	Pentane, cyclohexane	Open	
Olefins/alkenes	Octene, cyclohexene, isobutene	***Closed***	Olefins bind the catalyst tightly.
Aromatic hydrocarbons (halogen-free)	benzene, toluene, xylene	Open	No haloarenes
Ethers	Diethyl ether, THF, dioxane	Open	
Halogenated hydrocarbons	Dichloromethane, chloroform, chlorobenzene, bromobenzene	***Closed***	Halogenated solvents react with the Cu catalyst to make Cu–X bonds.
Protic	Water, alcohols (MeOH, EtOH, PhOH), carboxylic acids, thiols	***Closed***	React with catalyst similar to water.
Strong donor ligands / volatile bases	Phosphines, nitriles (incl. MeCN), pyridines, amines	***Closed***	Any reagent/solvent that is a good ligand for late metals will bind the catalyst.

Table 2. Guide to commonly used solvents.

Solvent	Purifier Open or Closed?
Pentane, hexane, cyclohexane, petroleum ether	Open
Benzene, toluene, xylene	Open
Diethyl ether, THF	Open
Dichloromethane, dichloroethane, chlorobenzene, fluorobenzene	***Closed***
Acetonitrile	***Closed***
Pyridine	***Closed***
Water, Methanol, Ethanol	***Closed***

Table 3. Purge times.

Reason for Purge	Length
Halogenated solvents (e.g., CH_2Cl_2, C_6H_5F)	10 min
Coordinating solvents (e.g., PMe_3, MeCN, PhCN)	15 min
Water, alcohols	15 min
O_2 spike (10-50 ppm)	10 min
O_2 spike (50-150 ppm)	20 min
O_2 spike (>150 ppm)	40 min (turn off O_2 analyzer)
Before use celite, sieves or alumina	15 min

C. Czar vs User Responsibilities

Communication and camaraderie are key to the successful operation of our gloveboxes. Czar-user communication should be a two-way street and "teams" of frequent box users should work together to support everyone's glovebox experiments. For example, czars should inform users of any maintenance updates, while users should communicate any problems they notice to their box lead and talk frequently with their czar and other users about any new procedures or reagents for that box. Frequent interactions among all the main users of the box and the czar are expected. Additionally, both users and czars are expected to be generous with their time, to communication openly, and to be willing to assist with troubleshooting. These are all aspects of a supportive training and mentoring culture, and are essential to gloveboxes operating at the highest possible level (and thus supporting high quality chemistry) . The responsibilities of czars and users are outlined below. These are guidelines for helping all group members achieve their experimental goals while also maintaining excellent glovebox operation, through the team mindset that is core to our group. ***<u>Group members who have concerns about glovebox culture or missed responsibilities should speak with Alex</u>***.

Czar Responsibilities:

- Lead and encourage box "team" communications
    - Check on the box frequently and be available to help users solve problems
    - Communicate with Alex and the other box czars
    - Communicate with users and encourage regular box "team" conversations
- Train new users and coordinate personal storage bins
- Box maintenance and troubleshooting:
    - Print log sheets and encourage their use
    - Dry alumina and sieves
    - Change pump oil
    - Clean out box shelves and freezer (and communicating with users beforehand)
    - Regenerate the box
    - Coordinate balance calibrations
    - Make atmosphere tests available for users
    - Lead troubleshooting concerns (i.e. noises, holes in gloves) with the users who report them to you (note: this could be a great training opportunity for someone who hasn't patched a glove!)
    - Communicate with Mbraun and Alex about any part maintenance / troubleshooting

User Responsibilities:

- Read this document! Get trained!
- Communicate with box czar and "team"
    - Attend and participate in box "team" conversations
    - Communicate concerns to czar and other box users
    - Report problems (i.e. noises, holes in gloves, etc.) to czar. Be generous in helping with troubleshooting and take advantage of training opportunities (i.e. learning how to patch a glove).
    - Ask questions!
- Communal box maintenance and personal supplies:
    - Check tank levels after box use and change empty tanks
    - Change vacuum trap pump oil if solvent is sucked into the pump
    - Refill solvent bottles
    - Change vacuum line if bump and communicate with other users
    - Leave no trace: use trays, clean up after yourself
    - Use the logbook
    - Restock consumable supplies (i.e. vials, caps, pipettes, tape, etc.)
    - Check the atmosphere
    - Contain personal chemicals to personal bins
    - Check for duplicates before you bring a new chemical in

    - Label all your chemicals
- Ask questions

D. Calendar and Logs

Users reserve time in the gloveboxes via online calendars (users obtain access to the calendars during training). The use of the Google calendars to schedule box time is critical, so that other users know when the box is in use and what solvents are in the atmosphere. To sign up for time, create an event in the appropriate glovebox calendar and title it with your initials, the solvents you'll be using, and any other pertinent information (i.e. AJMM: DCM, pentane, coldwell). If you see that another user has booked time using a compatible solvent, you're encouraged to share the glovebox time upon checking with that user. Out of the ordinary use or large chunks of time should be discussed with other frequent box users ahead of time. Lastly, if you edit or cancel your own time the day-of, notify other users via Slack. Working together is encouraged, and notes about cohabitation of the glovebox can be added to the calendar as needed.

Users indicate specific operations using paper logs located by the antechambers. Using the logs is essential! Other users need to know who is (or recently has been) using the box, whether the antechambers are in use, why the box is purging, among other information. Each user should consult the log before beginning use of the glovebox. For example, if someone has recently exposed the antechamber to air, refilling it with N_2 from the box without cycling could compromise the box atmosphere! Users must write an entry on the log each time the large or small antechamber is being used (L/S), if you're coming in or out of the box (I/O), if the box was purged (P), the time (plus times for the second and third cycles if coming into the large antechamber), if the task has been completed, if the tank level has been checked before purging, what stuff came in or out of the box, and any comments (i.e. what solvents were used and purged, if the tank was changed). An example is below. If the log is full, please print out more sheets. The Excel file is located in the group shared drive under Equipment and Software / Gloveboxes.

Date	Initial	L/S, I/O, P	Time	#2	#3	Done	Tank Checked	Stuff In/Out	Comments
07/15/2022	AJMM	SI	3:00 PM			X		vials	
		SOP	3:40 PM			X	X		MeCN

E. Noises

Gloveboxes make a variety of distinctive noises. Users should familiarize themselves with the common noises that a functional glovebox makes. Unfamiliar noises should be noted and traced to their source, as they are very often a symptom of a problem. See the Troubleshooting section for some examples of common problems (i.e. refilling more than usual if a vac port is open, clicking when a purge fails, etc.). Common sources of unusual noises include vacuum pumps, the circulation blower, pneumatic valves, and the refrigeration apparatus. If the problem cannot be detected and remedied, notify the box czar or Alex. Because of the importance of recognizing unusual noises, headphones must not be worn while working in the glovebox (as with all other lab work), and speakers should be kept at a reasonable level.

Note: With certain argon tanks, Phoenix makes a high-pitched squealing noise when it refills – a normal sound for this box that would be abnormal for any other.

F. PPE

Personal protective equipment for working in the glove box includes:

- Cotton gloves (only to be used for comfort with glovebox gloves)
- Safety glasses/goggles
- Sleeves (either long-sleeved shirts or separate compression sleeves used only for glovebox work)
- *Lab coats should be worn while bringing materials in/out of antechamber,* ***but lab coats should not be worn while placing arms inside the glovebox gloves****!*

Note: To handle reagents and glassware you're taking in or out of the antechambers or to handle the glovebox trap, change the cotton gloves to nitrile gloves.

G. Maintenance Logs

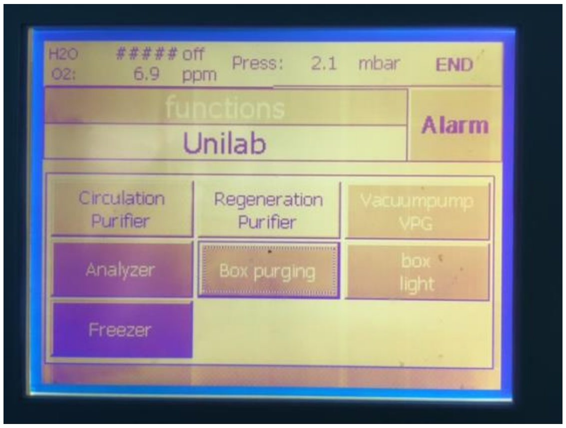
Each glovebox has its own maintenance log, usually attached to the side of the glovebox. All maintenance or repairs should be logged here by the box czar. If a user performs any maintenance they should communicate with the box czar and ensure that it is properly logged.

II. Computer

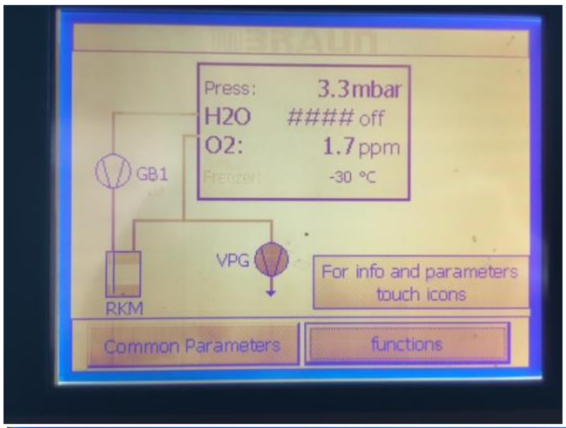
Each glovebox features a computer system that controls the major glovebox functions. This computer controls box pressure limits, the antechamber vacuum pump, the circulation purifier, the O_2 sensor, and is used to perform a regeneration of the purifier.

A. Operation

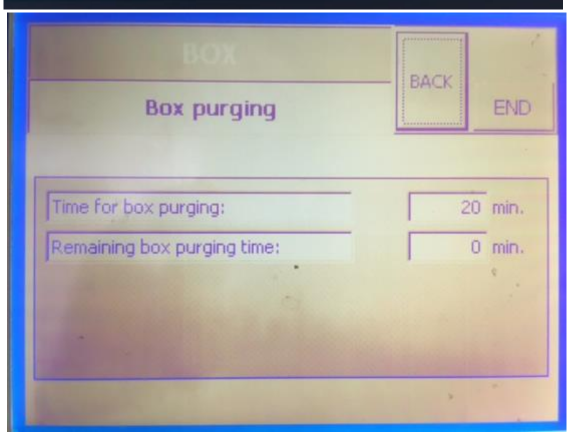
a)



b)

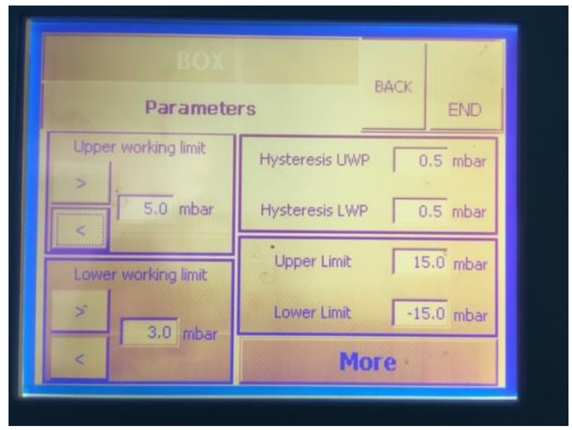


c)



d)

e)



f)

The glovebox computer has several features that are used on a regular basis. Image A represents the screen that is generally left up when the box is not in use. It indicates the status of the circulation purifier, the O_2 analyzer, the antechamber vacuum pump, and the box light. A dark grayed in box indicates that a function is ON. At the top of the screen the O_2 analyzer will indicate the O_2 level, which is accurate when the circulation purifier is turned on. The initial screen will also tell you the current working pressure of the glovebox. It is often useful to check the pressure during purges to ensure that it does not drop below 1.2 mbar.

This screen also controls box purging. The box can only be purged when the circulation purifier is turned off. Ensure that the circulation purifier is turned ON when the box is not in use. Note that the computer system will turn off the circulation purifier automatically if there is a rapid change in pressure (i.e. a person enters the glovebox too fast and spikes the pressure well above the upper working limit), so keep your eyes and ears open!

Be sure to carefully read each button before pressing it. Do not rush through screens or you may press a button that performs and unintended action.

If you hit END in the top right corner, you will bring up a secondary screen which will allow you to monitor certain glovebox parameters, such as working pressure, O_2 level, and the temperature of the freezer.

If you wish to return the initial screen from image B, you can click on the “functions” button. To edit certain parameters, like box purging time, then you can click on “Common Parameters” to bring up image D.

In general operation mode, you will likely only use the “Parameter Box Purging” tab in the bottom lefthand corner. This will allow you to edit the box purge time (image C).

This is where you can set the length of a purge consistent with the solvent that is currently in the atmosphere. (See Atmosphere section III).

If you suspect a box has been leaking, you may also press the “trends” button, which will bring up the O_2 trends for that glovebox over time.

B. Maintenance

Any changes to the glovebox computer for maintenance purposes should be discussed with the relevant box czar first and recorded in the glovebox notebook.

The glovebox computer contains several features that are useful for glovebox maintenance. These features are used much less commonly.

On the main screen (see the first image under “Operation”), the central box between circulation purifier and vacuum pump controls the “regeneration purifier.” This button is used to regenerate the glovebox circulation purifier, and should only be pressed when the full regeneration apparatus is set up AND the regeneration gas flow has been checked by the flow meter (See Regen SOP). Be careful NOT to press this button during normal operation, as the computer system will pass the point of no return quickly, and you may be forced to set up an unplanned regen. There is a confirmation screen that pops up before a regen starts, so be sure not to rush through the confirmation screen.

If you notice a change in the operational pressure in the glovebox (e.g. the glovebox pressure is working at less than 3.0 mbar or greater than 7.0 mbar), you may need to change the working pressure limits for the glovebox. This can be accomplished first be pressing the "END" button on any screen to bring up the following image (image B).

Clicking on the top square box that contains the pressure information (and the O_2 level) will allow you to edit some of the working parameters for the box.

Image E shows the upper and lower working pressure limits on the lefthand side of the screen. The box will refill when below the lower limit and exhaust when above the upper limit. The lower working limit should not be below 3.0 mbar. The upper limit should not exceed 7.0 mbar, but may be set slightly lower.

Additional parameters may be changed by clicking on the "More" button, but this is much less common.

III. Atmosphere

A. Operation

1. Introduction

The glovebox is equipped with an activated catalyst bed (a.k.a. purifier) which can remove O_2 and H_2O to levels below 0.1 ppm. The catalyst bed (purifier) contains a copper-based oxygen-reducing agent and molecular sieves. Copper catalyst removes oxygen by converting copper to copper oxide (CuO) while molecular sieves (10 Å) remove water and other small organic solvents. In normal operation the purifier is recirculating the N_2 atmosphere of the glovebox over the catalyst bed repeatedly. This means that N_2 in the glovebox should be improving over time, as more O_2 and H_2O are removed from the atmosphere with each pass over the catalyst bed. A good catalyst is essential to maintaining a healthy glovebox.

2. Taking care of the box atmosphere

Users should help maintain the efficacy of the catalyst and preserve the quality of the atmosphere by using care when working in the glovebox:

- Only use the solvents compatible with the catalyst bed when the purifier is operating, operate the antechambers as prescribed above, and use care when incompatible solvents or chemicals must be used in the glovebox.
- Test solvent with ketyl radical indicator to avoid oxygen or moisture contamination.
- Please note that all glassware should be taken out after use (please clean up after yourself)

- Never leave any glassware (pipettes, vials), Kimwipes with solvent in it after use.
- Make sure that the pump is on before opening any of the vacuum valves.

Some additional detail on solvent is provided here, and reproduced in the supplies section below. Only certain solvents will be allowed to be open at the same time. These "good" solvents include pentane, diethyl ether, tetrahydrofuran, benzene, and toluene. "Good" solvents may be used while the catalyst is open and can be opened at the same time or one after another without purging. Chemicals in the same class, that are not common solvents, such as hexanes, dioxane, or xylenes, can be used with the same guidelines. If you are not sure whether a solvent is "good" or "bad" please speak with the box czar. Oxidizing, coordinating, or otherwise damaging solvents are considered "bad" solvents. These solvents have the ability to a) reduce catalyst lifetime and b) react unfavorably with other users' compounds. Therefore, when methylene chloride, acetonitrile, alcohols, amines, water, or other coordinating volatile reagents are used, the catalyst must be closed, and the solvents must be kept isolated from each other, and especially the "good" solvents. Under no circumstances should a "good" solvent be opened when the atmosphere is contaminated with a "bad" solvent. If a user wishes to use these two classes of solvents at the same time, it is recommended that the user keep a 20 mL vial filled with the "good" solvent, only to be used when bad solvents are in use. When a "bad" solvent is in use, the user should make a public note of that on the Google calendar and logbook, so that other users do not unwittingly open their chemicals when the atmosphere is bad. Finally, the freezers should not be opened when "bad" solvents are in the atmosphere.

Check the atmospheric oxygen levels regularly by looking at the O_2 analyzer readout on the touch screen. O_2 levels should not be above 20 ppm during regular use. If the O_2 level spikes above 5 ppm, try to determine what caused the spike, and purge as appropriate to reduce the O_2 level. Note the O_2 level and (if known) what caused the spike in the log. If the problem is persistent and the cause is not readily apparent, notify the box czar or Alex to start an investigation.

Reason behind O_2 spike:

- antechamber not fully pumped down
- hole in gloves
- contamination of gas supply
- leakage in the tubing
- leakage through the antechamber: When the temperature of the room is too hot or too cold O-rings or seals of the antechamber might get too stiff or too soft which causes leakage in the antechamber.
- Deterioration of O_2 sensors: either needs calibration or change O_2 sensors.

What can you do if you find this problem?

- Purge (see *Table 3* in the Introduction) to reduce the O_2 level.
- Check the purity of the atmosphere using different chemical tests.

- Testing solvent for water impurities with ketyl radical indicator (for procedure check Chemicals and Supplies section)
- Use of a titanium metallocene as a colorimetric indicator for O_2: $[TiCp_2(NCCH_3)_2]^+$ can be used as a colorimetric indicator for oxygen contamination in inert-atmosphere glove boxes. The blue solution is conveniently prepared by dissolving small (mg) amounts of $TiCp_2Cl_2$ in acetonitrile in a scintillation vial, adding zinc dust, and capping. Aliquots of this solution may be removed by disposable glass pipet and transferred to an open vial. Inspection of the aliquot gives a qualitative indication of the dioxygen level in the glove box. As the solution is exposed to trace O_2 the solution color changes from blue to green and ultimately, under higher dioxygen concentrations, to yellow. [Reference: *J. of Chem. Ed*. **1998**, *75*, 459]

- Regeneration of the catalyst (see Regeneration under the Maintenance section).
- If regeneration failed to reactivate catalyst, the catalyst will need to be changed.

3. Purging

Preserving the glovebox atmosphere free from O_2 and moisture is important to maintain the longevity of catalyst/ purifier. Generally, the partial evaporation of solvents inside the box can contaminate other chemicals stored in the box. However, organic solvents can be removed by purging of the box with a positive pressure of inert gas and thus gloveboxes maintain their inert atmosphere. The box should be purged only if the atmosphere is contaminated (do not purge it without a purpose, you will only waste N_2). A researcher using “bad” solvents (see above) or other chemicals that would react with the Cu-catalyst (e.g., volatile acids and phosphines, amines, chlorinated solvents) is responsible for closing the catalyst bed during use and purging the glovebox thoroughly after contaminating the atmosphere. The table at the beginning of this document can be used as a guide, but purge time should be adjusted based on the amount of contamination (i.e., how much solvent was used and for how long). Suggested purge times are written above (see Table 3 in the Introduction).

The glovebox can be used during a purge. The N_2 regulator should not need to be adjusted during purges (or at any other time). However, it is possible that the problems in the N_2 supply (bad tank, pressure drop, etc) could cause over or under-pressurization of the box. Therefore, it is essential that users pay attention to purges and listen for clicks and other sounds that may indicate a problem.

4. Pressure limits

Recommended ***working*** *pressure limits:*

2 – 6 mbar.

Avoid negative pressure, mostly commonly encountered when the large antechamber is filled too quickly. Likewise, avoid large positive pressure, as this could lead to breaking the plexiglass or the gloves popping off! Enter the glovebox slowly to provide time for the pressure to equalize as you enter. High pressure could also be due to a malfunction of the footswitch or the electronic controller. Use the footswitch when necessary; it is a good idea to use the footswitch to raise pressure when filling the large antechamber. In cases of emergency: if the glovebox is over pressurizing, and the foot pedal is not working, refill the large antechamber, which will take N_2 out of the glovebox and into the chamber, lowering overall pressure. Seek the cause quickly, as this is only a temporary fix. If the box is underpressurized, use the foot pedal, or insert your hands into the box to increase the pressure. Check that the N_2 tank is not empty.

5. Purification and Circulation

The purifier is controlled by the touch screen (under "functions", see Computer section). When the purifier button is shaded, it is on, as further indicated by an audible whirring sound. Press the button to turn off the circulation and close the catalyst bed / purifier. The purifier should always be closed when using "bad" chemicals (chlorinated and protic reagents, amines, phosphines, etc). After using "bad" chemicals, the glovebox should be purged before the purifier is opened.

B. Maintenance:

1. Pump maintenance

Pump oil in all glovebox pumps should be changed every six months (at every group cleanup day) by box czars. Pump oil in the antechamber pump should also be changed after every glovebox regeneration. Of note, if pump oil is contaminated with solvent by accident, it needs to be changed by the responsible party.

2. Regeneration

Regeneration should be performed at least every 6 months, or when the purifier fails to maintain < 5 ppm O_2. Regeneration is performed with 95/5 N_2/H_2 mixture and takes about 800 psi of gas to regenerate the catalyst. The pump oil must be changed after regeneration. See the Glovebox Regeneration SOP for further information and instructions. If regeneration does not work, it is possible that something when wrong in the regeneration (e.g. the cylinder was empty); sometimes the catalyst bed itself needs to be replaced (approximately every 10 years).

3. Leaks

Avoiding leaks is an important factor to keep the glove box atmosphere in good condition, hence, the detection of leaks is important. An efficient way to detect leaks is to pay attention on how frequently the box is refilled, and if the consumption of gas has been increasing. When there is a suspicion of leakage it is usually located in:

- Gloves: therefore, must be the first place where you need to look for leaks.

- O-rings of the antechambers: the way to test is by evacuating the antechamber and leaving the valve in close for 1h (or more time if it is possible) if the pressure inside of the antechamber increases the O-rings are not working and is necessary replace them

- Connections and caps: If after looking for leaks in gloves and antechambers, suspicion of leaks persists, the next step is looking for the leaks in the connections (in the pipes) and in the caps (behind the glove box) with soap and water solution.

IV. Gloves:

Glovebox gloves are permanently installed in the box, and workers put their hands in the gloves from outside the box. Using these gloves, workers can reach all areas of the box. Generally, these gloves are thick and thus provide better protection while handling sensitive materials inside the box. These gloves can work at a temperature range from -40˚C to +90 ˚C.

A. Operation:

- Jewelry (watches, rings, bracelets, etc), can damage the gloves and must be removed.
- Clean gloves must be worn before inserting hands into Glove box gloves. Users may opt to keep a pair of clean cotton gloves to reuse for this purpose.
- Care must be taken in using sharps, scissors, forceps, glass pieces, etc. which could damage the gloves.
- Minimum use of solvents (benzonitrile, THF) as it can also destroy the rubber material of gloves.
- Gloves should be cleaned by kimwipes after every reaction.

B. Maintenance:

- If you find hole in gloves, offer to help the box czar to fix it either by attaching bicycle patches or by changing gloves. Signs of a punctured gloves may be visible tears, smelling solvents, or feeling cold spots.
- For changing gloves, one can look at this link https://seabaugh.nd.edu/assets/330934/at_man_mbraunglobebox_mb20_200_lmsp_dp_std_en.pdf (page number: 138 to 141) and follow the procedure.

V. Antechambers

A. Operation

Always check to see if the antechambers were recently opened to air. Be sure to fully evacuate the antechambers before refilling them, otherwise the glovebox atmosphere will be contaminated. Always bring materials into the antechambers on a tray to avoid making a mess in case of a spill and be mindful of how your items are placed and how gently you fill and evacuate the antechamber, since things tend to move around during the cycles. Don't worry about drying items that will be removed quickly from the glovebox (like trays).

Small antechamber:

1. First ensure that the valve on the **large** antechamber is closed to avoid contaminating its headspace when you open the small antechamber to vacuum.
2. Fully refill the small antechamber and turn the valve to the "closed" position. Now the outer door can be opened and vacuum-ready objects (see "bringing in chemicals" section below) can be safely loaded onto the tray.
3. Close the door and turn the valve to the "vacuum" position. Five (5) evacuate/refill cycles should be performed, with each evacuation lasting for at least 30 seconds after reaching full vacuum.
4. After refilling the antechamber on the last cycle, point the valve to the "closed" position. Now the inner door can be opened, and the items inside can be unloaded from the tray.
5. After bringing everything into the glovebox, close the inner door and evacuate the small chamber reopen the valve on the large antechamber to return it to dynamic vacuum.
6. To remove objects from the glovebox, first ensure that the antechamber has been sufficiently evacuated since being exposed to air. Then, refill the chamber, turn the valve to "closed" position, and open the inner door. Insert items to be removed from the box, close the door, and then open the outer door. Remove items, close door, and evacuate the chamber.

Large antechamber:

1. First ensure that the valve on the **small** antechamber is closed.

2. Fully refill the large antechamber and turn the valve to the "closed" position. Now the outer door can be opened and vacuum-ready objects can be safely loaded onto the tray.
3. Close the door and turn the valve to the "vacuum" position. Three (3) evacuate/refill cycles, should be performed, with each evacuation lasting 20 minutes after reaching full vacuum.
4. After refilling the antechamber on the last cycle, point the valve to the "closed" position. Now the inner door can be opened, and the items inside can be unloaded from the tray.
5. To bring items out of the glovebox, make sure the large antechamber has been evacuated sufficiently since being exposed to air. If the large chamber was under *static* vacuum (valve in "closed" position), partially refill the antechamber, then evacuate to full dynamic vacuum for a few minutes.

Overnight evacuation: Anything that will be stored in the box long-term that has not been oven-dried, such as plastic, paper, wood, or rubber should be pumped into the box overnight. This includes electronics (fans, stir plates) and especially paper products which contain a lot of water. Kim wipes should be heated to 60 ºC for 48 hours before pumping in overnight.

B. Maintenance

It is important to observe the logbook and be aware of recent antechamber activity. Remember that there is only one pump connected to both the large and small antechambers. Consider that if the chambers are not isolated from one another, air introduced to one chamber can be exchanged into the other. The chambers should not be left under static vacuum or atmospheric pressure, as leaks could develop. One way to notice a leak in one of the antechambers is to evacuate the chamber and then leave it on static vacuum. An increase in pressure indicates a leak. The solution is **very rarely** tightening either the inside or outside door, and in fact overtightening either door should be avoided. Often replacing the o-ring on one of the doors will solve the problem, unless there is another damaged part that should be promptly replaced. Users should check regularly for leaks, and the box czar should ensure that there are no leaks during every group cleanup day. Another reason for the antechambers not fully pumping down could be a spill or a leak in a vessel being brought in. Whenever you notice the antechambers struggling to reach a full vacuum when bringing items into the glovebox, it is important to stop immediately and assess the situation.

VI. Group Supplies

While some supplies are provided by the czar (outlined below), all other supplies (e.g. solvents, vials, etc.) must be replaced by users. If you notice a czar-provided supply is running low, please inform the czar. Consider a simple act of kindness: offering to help replenish the supply for your labmates!

Reusable tools: Spatulas, pipet bulbs, etc.

The common tools for inert atmosphere glovebox reaction chemistry should be present in the glovebox for general use. Reusable tools such as spatulas, pipet bulbs, and filtration devices should be replaced where they belong and kept clean. At least two of each common tool should be available to facilitate working together; if you think something might be missing, consult the box czar.

Disposables: Vials, Pipets, etc.

Disposable items include vials (20 and 4 mL), vial caps, pipets (long and short), glass microfiber filters, NMR caps, kimwipes, crystallography slides, and grease. As the supply dwindles, users should replenish the items. Be a good lab citizen and make sure you are contributing to your box by regularly bringing in new supplies! If you are not sure where the supply is or how best to bring it in, consult the czar. Replacement vials and pipets should be kept in the oven, ready to be pumped into the box. Glass microfiber filters should be pumped in overnight, after being warmed in the oven for more than 24 hours. Kimwipes should be warmed in the small warming oven, for 2-3 days; then, they are to be pumped on for 24 hours in the large antechamber. Leaving kimwipes to pump in during the weekend is best practice. Kimwipes retain an incredible amount of water.

Celite / Sieves / Alumina

Celite is a trademarked name for diatomaceous earth (fossilized remains of single-celled alga called diatoms), a filtration aid. It is highly recommended for removing fine precipitates such as silver halides. Molecular sieves are uniformly porous alumino-silicate materials that are generally used in our lab as desiccants, with 3 Å and 4 Å sieves both absorbing water effectively. Alumina (aluminum oxide) is used as a desiccant or for chromatography. These staple chemicals are activated/dried by the czar by heating to 200 °C overnight under vacuum (see the Drying Alumina/Celite/Sieves SOP for further instructions). Due to the variety of coordinating solvents used in the glovebox, the ***utmost care*** should be taken to avoid exposing these containers to ***any*** solvents (see Solvents section). A purge time of 15 minutes should be performed before opening containers if solvents have been introduced to the atmosphere. All trash (pipettes, vials, etc.) contaminated with solvents should be removed from the box prior to opening. Plan ahead: take the celite you need before you start a reaction with any solvent. Consider carefully what is in the atmosphere before opening these canisters, so that they remain useful tools for the group. If contaminated, you and your fellow groupmates' chemistry will be compromised.

Solvents

Solvents available for group use include pentane, diethyl ether, tetrahydrofuran, benzene, toluene, methylene chloride, and acetonitrile. Solvents will be stored in 1 L glass bottles equipped with activated 3 Å molecular sieves. Sieves in solvent bottles should be replaced by czar every group clean up, and the date of the last replacement must be written on each bottle. Users are responsible for bringing solvents into the box, and refilling solvent bottles that are almost empty. If a user plans on using a large (> 50 mL) amount of solvent, the user should bring in their own solvent, and not use group supply. Solvents taken from the activated alumina solvent columns should be briefly degassed, and then tested with a purple solution of sodium benzophenone ketyl radical (except halogentated and coordinating solvents). See ketyl radical recipe in the Appendix.

Procedure for testing solvents: Fill a 4 mL vial with solvent. Add one drop of ketyl solution from a disposable glass pipet, and stir/shake the vial. If the solution stays purple, it is dry. If it goes colorless or blue, add one drop at a time until it turns purple. The general guidelines for dryness are as follows: pentane, benzene, and toluene, no more than 1 drop to purple; diethyl ether, up to 2 drops to purple; THF up to 3 drops to purple. If a solvent does not pass, try degassing more, then testing again (because oxygen also reacts with the ketyl radical). If it still does not pass, the solvent can be run through alumina in the box and tested again. If the test indicates that the solvent is grossly wet, remove the solvent from the box and talk to the solvent column czar. The user bringing in the solvent is responsible for the solvent being dry. If it is not dry, do not leave it in the box! Note that color changes to green, orange, or yellow are indicative of chlorinated solvent, and may indicate a contaminated solvent or atmosphere.

In order to keep the atmosphere clean, and to maintain a box that is suitable for everyone, only certain solvents will be allowed to be open at the same time. These “good” solvents include pentane, diethyl ether, tetrahydrofuran, benzene, and toluene. "Good” solvents may be used while the catalyst is open and can be opened at the same time or one after another without purging. Chemicals in the same class, that are not common solvents, such as hexanes, dioxane, or xylenes, can be used with the same guidelines. If you are not sure whether a solvent is “good” or “bad” please speak with the box czar. Oxidizing, coordinating, or otherwise damaging solvents are considered “bad” solvents. These solvents have the ability to a) reduce catalyst lifetime and b) react unfavorably with other users’ compounds. Therefore, when methylene chloride, acetonitrile, alcohols, amines, water, or other coordinating volatile reagents are used, the catalyst must be closed, and the solvents must be kept isolated from each other, and especially the “good” solvents. Under no circumstances should a “good” solvent be opened when the atmosphere is contaminated with a “bad” solvent. If a user wishes to use these two classes of solvents at the same time, it is recommended that the user keep a 20 mL vial filled with the “good” solvent, only to be used when bad solvents are in use. When a “bad” solvent is in use, the user should make a public note of that on the Google calendar and logbook, so that other users do not unwittingly open their chemicals when the atmosphere is bad. Finally, the freezers should not be opened when “bad” solvents are in the atmosphere.

Deuterated Solvents

All deuterated solvents should be dried on a personal need basis and kept in personal storage. Remember that deuterated solvents react the same as their proteo analogues, so CD_2Cl_2 is a “bad” solvent and requires the catalyst to be closed during use and a purge after use. To avoid contamination with any solvents introduced to the box atmosphere prior to use, a purge time of at least 10 minutes is required.

Trash

It is each user’s responsibility to clean up after themselves and maintain a clean glovebox. The most efficient method to keeping the box clean is to keep all trash generated during your time in an aluminum tray. At the end of your time, the tray can be pumped out and contents can be disposed of appropriately. Contaminated glassware and Kimwipes should be brought out after use, and before the “after bad solvent” purge. Best practice is to bring in an aluminum tray that has been warmed in the large oven overnight during the start of your time. However, a tray that has not been warmed can still be used if brought out immediately after you are done working in the glovebox. Pyrophoric or toxic waste generated inside the box needs to be removed immediately as well.

User Chemicals

Each user will need various chemical reagents during the course of their studies. Both commercial reagents and synthesized materials may be brought into and stored into the glovebox as needed.

Solids: To bring in dry, air exposed solids, cover the container with kimwipe secured with a rubber band. Make sure the solids are not prone to sublimation (e.g., ferrocene, Co_2CO_8); if they are a sublimation risk, degas in a Schlenk flask cooled with dry ice/acetone, and bring the sealed vessel into the box. Reactions run in Schlenk flasks can be dried under vacuum to leave a solid residue before being brought into the box. Vials or glass bottles from other boxes should be tightly sealed with electrical taped before exposure to air. These bottles can be usually be safely evacuated (although beware odd-shaped glassware or very large bottles).

Liquids: Purify and degas liquids by standard procedures and store outside of the box in a Teflon-stoppered flask (no ground glass stoppers!). Vessels containing liquids can be pumped in under pressure or vacuum if a Teflon-stoppered vessel (e.g., “bomb” or “Straus flask”) is used. Do not use Schlenk flasks for bringing liquids in the box. To bring in Sure-Seal bottles, make sure the seal is not punctured, seal the cap with electrical tape, then pump in the bottle, turn off the purifier, take the cap off, purge, and turn on purifier.

All chemicals should be labeled with the date when they were opened (in or out of the box), the date they were brought into the box, and the initials of the person bringing them in. Chemicals that are going to be a risk to the box (acids, chlorinated, etc) should be taped tightly at all times, to prevent leakage and contamination.

Storage

In order to allow efficient storage, stackable containers should be used. Chemicals should be stored on the shelves rather than on the floor, if possible (exceptions: large or unwieldy bottles and mercury). Containers with tools and materials for general use (spatulas of various types, grease, tape, caps of various sizes, stir bars, rubber stoppers, etc.) will be available. The box czar will make sure that these materials are available and cleaned during box cleanup.

Any materials for personal use should be stored in personal storage space. Chemicals that are sensitive to common atmospheric contaminants should be well taped. Chemicals that are themselves atmospheric contaminants should be well taped to prevent leakage into the atmosphere. As a courtesy to others, do not store large equipment or reaction apparatus on the floor for extended periods of time.

Inventory

Shared glovebox chemicals will be entered into the group chemical inventory spreadsheet. If you empty a bottle, make sure it is removed from the inventory. If you bring in a new bottle for general group use, make sure it is added to the inventory (or moved from its old location).

VII. Balances

The correct use of the balances is essential because a small error can change research results.

A. Operation

Before using the balances, it is important to check that it is level; the bubble of the level should be in the middle of the circle.

To ensure accuracy:

- Tare the balance before using it
- Tare the empty vial before weighing out material
- Close the balance doors and wait for stabilization for each measurement
- Weight at least 10 mg
- Verify the weighed mass at least twice
- Try to keep the pressure inside of the box stable during the weight
- To avoid eccentricity problems, the vials have to be in the center of the weighing pan

Respect the limits of the balance and remember that when working close to the upper limit, the accuracy of the balance decreases and measurement errors may occur. Keep the balance clean after using it.

B. Maintenance

In general, it is important to keep the balance clean for good performance; one of the areas of the balance in which special care must be taken is in the weighing pan because this is the area where reagents can remain and subsequently damage. It is important after using it, to make sure that no reagents remain and to clean any residue off with a brush.

It is important to avoid moving the balance from one place to another in order to keep it level and not affect its calibration.

The scales must be calibrated every year, updating the date of the last calibration. The maintenance contact and calibration data should be kept close to the balance, accessible for both czars and users.

C. Glovebox-specific notes

Static inside the box can affect the measurements, so if static problems occur, or the weight does not stabilize, the use of an anti-static gun is recommended. Check with the box czar to see if these static problems correlate to poor condition of the box atmosphere.

VIII. Coldwell and Freezers

A. Operation

1. Freezers

Each glovebox has a -30 ºC freezer. Inside each freezer, there is a secondary thermometer which can be used to confirm freezer temperature displayed by the glovebox computer. The freezer has four shelves of adjustable height. The top two shelves should be used to store shared chemicals, while the bottom two shelves are designated for storing personal chemicals or crystallizations. Bottles stored in the freezers should be tightly capped to avoid leakage or cross-contamination. Special care should also be taken when cooling Teflon-sealed glassware in the freezer, since Teflon and glass have different thermal expansion coefficients, which can lead to seals breaking upon cooling and leakage. It should also be noted that some solvents (benzene, DMSO, water, etc.) will freeze at -30 ºC; do not attempt crystallization in these solvents in the freezer and use caution in other applications to avoid breaking glassware due to solvent expansion upon freezing. ***Freezers should not be opened if the box atmosphere is contaminated with a "bad" chemical*** (moisture, acid, chlorinated solvents, amines, phosphines, etc.). If the freezer gets contaminated, the box should be purged with the freezer open. Be careful when accessing your chemicals, as your labmates may have crystallizations set up in the freezer. Label bottles carefully, and use paper and tape for labeling items that will be stored for long periods, as Sharpie marker labels often wear off in the freezer over time.

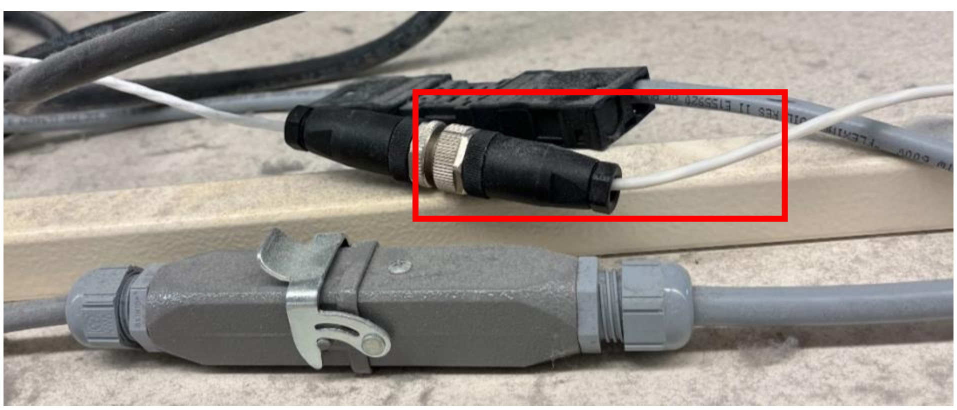
2. Cold well

The cold well can be used to reach temperatures below -30 ºC inside the glovebox, though it should be noted that better temperature control can be obtained in airtight glassware on the Schlenk line.

To set up the cold well, first ensure there is a dedicated stir plate underneath it. It may take some patience to situate the stir plate in a position to stir your reaction, but this step is vital to ensuring rapid temperature equilibration upon additions to your chilled reaction mixture. Next, the cold bath should be prepared. Using the chiller and isopropanol allows cooling to -80 ºC, and use of liquid nitrogen allows cooling to -196 ºC. *Do not use liquid nitrogen as a cooling agent in a glovebox filled with argon!* The inert argon atmosphere will condense into the cold well, causing large and dangerous pressure fluctuations. Other temperatures can be reached using other cold baths, but it is usually more convenient to carry out such procedures on the Schlenk line. To ensure that the cold well is reaching your desired temperature, the thermometer usually stored inside of the freezer should be removed and placed in the cold well. Stir plates should not be lowered in the cold well because electrical cords will easily crack due to the low temperature. A stir plate should always be used on the outside of the cold well. Extra caution should be exerted when performing potentially exothermic reactions – even though the reaction is started at low temperature, nitrogen gas is not a good heat transfer agent, so the temperature of a reaction mixture has the potential to increase rapidly. If a "bad" chemical is used in the cold well, uncap the cold well and allow it to warm to room temperature (using a heat gun if needed) before purging. If these steps need to be taken, record them in on the glovebox log and note them on the calendar to inform your boxmates.

B. Maintenance

No preventative maintenance on either the freezer or cold well should be needed. The freezer should be inventoried and cleaned during every group cleanup. Additionally, we have had problems where the freezer temperature reading is inaccurate and/or freezer temperature alarms show on the glovebox computer screen due to a loose electrical connection (also see Troubleshooting table in the Appendix). To fix this issue, locate and tighten the sensor connection on top of the glovebox (pictured below, red box indicates the connection to tighten).



C. Glovebox-specific notes

Freezer and cold well practices are shared between gloveboxes. If you encounter an issue with either that is not covered here, consult the box manager and document it in the glovebox log. **Warning: *Do not use liquid nitrogen as a cooling agent in a glovebox filled with argon (i.e. Phoenix)!*** The inert argon atmosphere will condense into the cold well, causing large and dangerous pressure fluctuations.

IX. Auxiliary Vacuum

A. Operation

The boxes are equipped with an auxiliary vacuum pump for filtrations and removing solvents. The rubber tubing can be attached directly to a side-arm flask, or to an adapter that can be affixed to 20 mL vials. The first user to need vacuum should set up the trap, cooled with liquid N_2. That user will be responsible for the trap for the day, until responsibility is passed on to another user, or the trap is dropped. Responsibility is usually passed on when another user uses the trap after the original user is finished. Both users should acknowledge that responsibility for the trap has been passed on.

The auxiliary vacuum is attached to a vacuum gauge. There is a yellow tag on the gauge that specifies typical pressure readings. Before adding liquid nitrogen to the trap, be sure the system reaches the indicated pressure. If the system doesn't reach the usual pressure, it might indicate a leak in the auxiliary vacuum line. The source of the leak or high pressure reading should be solved before adding the N_2(L) to avoid condensate oxygen.

Before evacuating "bad" solvents or other chemicals that may contaminate other compounds that are under vacuum, one should consult with the other users of the box. In case they cannot be found, their containers should be closed to vacuum before pumping on contaminating mixtures.

To avoid contamination between uses, the Kimwipe in the vial attachment should be replaced with each use. If the vial attachment septum becomes contaminated, it should be replaced as well. Contamination of the septa and connected vacuum line can be reduced by avoiding "bumping". "Bumping" occurs when the vial contents abruptly splatter against the sides/mouth of the vial and may include vial contents getting into the vacuum tubing. Bumping is best avoided by opening containers to vacuum slowly, not disturbing solutions being pumped on, and minimizing the amount of solvent in a container being pumped on. A good rule of thumb would be to pull vacuum on vials that are half full at most. If bumping does occur, the contaminated attachments should be cleaned or replaced (see Maintenance).

Another key to normal operation of the vacuum line is ensuring that the septum/vial seal is good when pulling vacuum. The septum should fully cover the mouth of the vial so that it is making contact with the sides of the vial. The seal can be disrupted if the Kimwipe gets wedged between the rubber and glass, or by not pushing the septum down far enough. This will both slow down anything being dried on the line and cause the glovebox to refill frequently as nitrogen gets sucked through the pump, reducing the box pressure.

The last person to work in the box should check that the trap was taken down. All the valves inside the box and the valve next to the trap must be closed at the end of the day. If the trap is to be left up overnight, make sure the Dewar is completely full **of dry ice/IPA** before you leave, and remember to refill it in the morning.

B. Maintenance

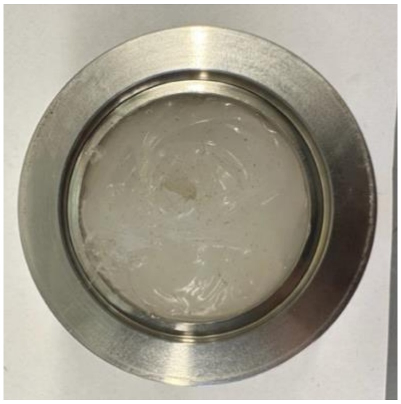
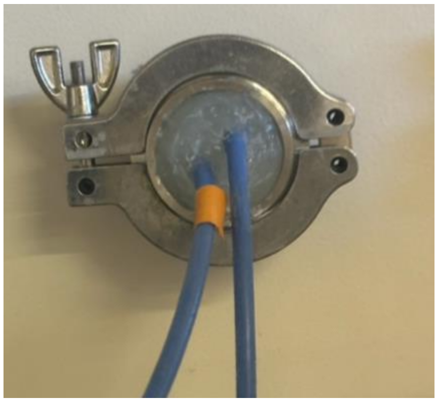
1. Septa

In order to make the holes in septa for vacuum ports, take the washer cutter, PTFE stand, and a hammer, and set them up as pictured. Simply hit the washer cutter with a hammer until you have bored a hole all the way through the septum and remove the washer cutter. Do NOT use needles as this is dangerous both for users and the box itself.

2. Building a Feedthrough

To build a feedthrough is necessary the use of flanged feedthrough (size KF-40), silicon, and the wire. it's necessary to fill the inside of the circle of the flanged with silicon trying to leave a hole in the middle to pass the wire or the wires (if you need to set several wires in the feedthrough be sure that the hole is big enough to put all the wires and silicon among those), before finish filling the circle and fixing each wire, make sure that the part of the wire that goes inside the box and the part that goes outside the box are in the right direction, as well as the length of the wire inside the box is correct.

NOTE: Once the feedthrough is set up in the box, is necessary to check leaks with snoop or soap and water.

To set up the feedthrough in the glove box, is necessary to remove one of the KF-40 caps and replace it with the feedthrough, be sure that you have all the necessary pieces to do it like the o-ring and the clamp. Once the feedthrough is installed purge the box for 20 min and check leaks.

3. Cleaning tubing

When bumping has occurred, any contaminated tubing and attachments should be cleaned or replaced. It is the responsibility of the person who bumped to do the replacement. Clean tubing should be kept in the warming oven, ready for use. Any tubing contaminated by bumping should be cleaned and placed into the warming oven for later use and a new (dry) piece of tubing brought in. The same applies to any glass attachments, although they should be placed in the higher temperature oven. Septa should be discarded and replaced. A supply of fresh septa should be maintained in the warming oven.

X. Feedthroughs

A. Operation

1. Electrochemistry

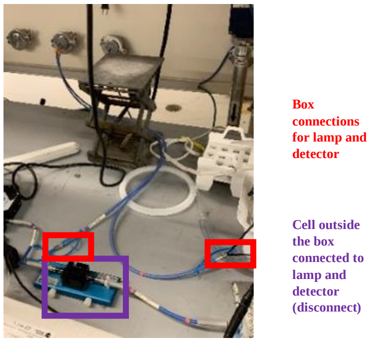
Electrochemistry experiments can be performed in gloveboxes either through a). electrode cables in the box connected to a feedthrough connection outside the box that can be connected to a Wavedriver potentiostat, or b). smaller Wavenow potentiostats and electrodes in the box that wirelessly connect to a laptop kept closeby. For the Wavedriver option, no additional cords will need to be brought into the box, as they are already fed into the box. Once the Wavedriver is connected to the box feedthrough and a laptop, electrochemistry experiments can begin. For using the Wavenow potentiostats, these smaller potentiostats and their power cords and electrode connections will need to be pumped into the box. Wavenow potentiostats can be pumped into the small antechamber of the box through 25-30 cycles *slowly* going to only ~75% vacuum and then immediately refilling (again, slowly). This is out of caution to the electrical connections in the potentiostat. To remove any oxygen brought into the box by forgoing complete vacuum cycles, the box should be purged for 30 minutes after bringing the potentiostat in. This setup can then be assembled in the box, and the electrochemistry experiment can be controlled using a laptop wirelessly connected to the potentiostat through the Wavenow USB drive that matches the barcode listed on the potentiostat. This flashdrive will flash blue when connected properly. However, even if the laptop is "connected," spikes may appear in your data if it's too far away from the potentiostat. For both the Wavenow and Wavedriver, electrode connections are as follows: gray = ground, red = working, green = counter, and white = reference. More information on electrochemistry experiments can be found in the Electrochemistry_CV and Electrochemistry_CPE SOPs.

When your experiment is completed, be sure to hang electrode cords off the floor of the box so that they are out of the way of other users and to minimize the risk of pinching the cords. Similarly, be wary of pinching the electrode cords when using the antechambers doors, as this will both damage the cords and create a leak in the box.

2. UV-vis

UV-vis experiments may be performed in cuvettes in the glovebox placed in a sample holder in the box. The sample holder is connected to two fiberoptic cables that are fed through the box to the Ocean Optics UV-vis spectrometer. To set up the experiment in the box, the sample holder will need to be connected to the two blue fiberoptic cables in the glovebox and the instrument will need to be connected on the bench to the opposite ends of these cables that come out of the box feedthrough. Ensure that one cable leads to the lamp and the other leads to the detector on the benchtop. If the sample holder outside the box is connected to the lamp and detector, disconnect and cap those cables and connect the cables from the box feedthrough. See below.

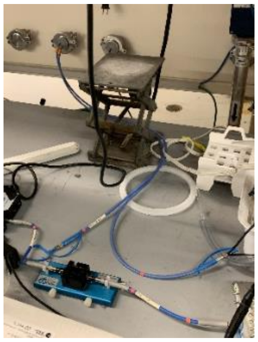
Sample holder setup inside the box



Cable connections outside the box

Users should be wary of pinching or bending these cables as they can be easily damaged. The fiberoptic cables should be capped when not in use and stored out of the way (see below), being careful to leave them curved at their natural arc. For more information on setting up an UV-vis experiment, see the UV-VisOceanOptics SOP.

Cable storage inside the box



Cable storage outside the box

3. Power strips

Power strips are located in all gloveboxes towards the back. They should remain on during routine use of the box, and the box czar should be informed if there is a power issue or a pinched cord. Similarly, if you turn off the power strip or unplug any cords in order to minimize electrical noise during an electrochemistry experiment, be sure to turn all power back on after your experiment so that other users may resume routine operation of the box. If moving equipment around the box, be wary of pinching the power strip cords.

B. Maintenance

In maintenance part 2, is described how to build feedthroughs.

With the continuous move of the wires and it use inside of the glove box, With the continuous movement of the wires and their use inside the glove box, the feedthroughs could have leaks, test the leaks with snoop or soapy water.

XI. Appendices

A. Part Numbers

If there is a part that you end up ordering that does not appear on this list, please add it! Questions about how to source specific glovebox parts should be directed to MBraun Customer Support, (603) 773-9333.

Item	Vendor	Part Number
Alumina	VWR	EM-AX0612-1
Anti-Static Gun	Fisher Scientific	NC9663078
Baby Powder	Johnson's / Amazon	Amazon Link
Benzophenone	Sigma-Aldrich	B9300
Bins for Storage	Global Industrial	WB269681BL
Catalyst for Gas Purifier	MBRAUN	Copper Catalyst • MB10 Gas Purifier
Celite	Sigma-Aldrich	22140
Fan	Holmes / Amazon	HNF0410A-BM
Flow Meter	MBRAUN	Flowmeter
Glass Adapters for Vac Line	Chemglass	CG-1014-14
Glove O-Ring	MBRAUN	2600239
Glove Liners	Fisher Scientific	14-379
Glovebox Gloves	Grainger MBraun	4T479 Honeywell 8N1532A/9Q
Glovebox Gloves	Honeywell	8N1532A/9Q: Neoprene Glove Box Gloves, Honeywell Safety, Ambidextrous, Diameter = 8, Glove Color= Black, Length = 81.3cm (31 7 / 8), Thickness = 15mill. Glove Size= 9.75
HVAC Filters	MBRAUN	MBRAUN Fine Filter
Internal Glove Port Cover	MBRAUN	MBRAUN 8" Internal Glove Port Cover
KF-40 Caps	Amazon	KF-40 caps

Large Antechamber O-Ring	MBRAUN	MBRAUN Phoenix: OR-01 Other Boxes: 2400309
Pump Oil	VWR	54996-082
Septa for Vac Line	Chemglass	CG-3024-05
Sieves for Catalyst	MBRAUN	3240262
Sieves for Solvent Drying	Sigma-Aldrich	MX1583
Sleeves	Keeble Outlets / Amazon	Amazon Link
Small Antechamber O-Ring	MBRAUN	7077297
Sodium Metal	Sigma-Aldrich	282065
Solvent Bottles	Qorpak	GLA-00817
Solvent Bottle Caps	Qorpak	CAP-00191-EA
Tape (Black electrical)	Scotch / Staples	23978171
Thermometer	Chemglass	CG-3503-L-20
$Ti(Cp)_2Cl_2$	Sigma-Aldrich	234826
Vacuum Tubing	VWR	60985-534
Workstation Ionizer	MBRAUN	Workstation ionizer
Zn Powder	Sigma-Aldrich	1087890500

B. Ketyl Radical test

Benzophenone Ketyl Radical Recipe (Courtesy: Prof. Theo Agapie, Caltech)

Recipe: In the glovebox, weigh 28 mg (1.22 mmol, 1.6 equiv) sodium metal into a 20 mL scintillation vial. Add 0.137 g (0.752 mmol) benzophenone (Ph_2CO), 20 mL THF, and a magnetic stir bar. Stir vigorously overnight. The clear, colorless solution should darken steadily to a blue color before eventually turning inky purple. The excess sodium will keep the indicating solution active and accurate for longer.

Derivation:
We want to test for < 10 ppm water in our solvents. What concentration of ketyl radical do we need?

For any solvent, we can choose the following parameters:
Desired maximum level of water impurity: $p \times 10^{-6}$ (p ppm)
$d_{solvent}$ = density of solvent to be tested
$FW_{solvent}$ = formula weight of solvent to be tested
FW_{Ph2CO} = 182 g mol^{-1} = formula weight of benzophenone indicator
Volume of solvent to be tested: V mL (normally 4 mL – full small vial)

Moles of solvent = $(V \times d_{solvent}) / FW_{solvent}$

⇨ Max moles of water = $(p \times V \times d_{solvent}) / FW_{solvent}$

Prepare 20 mL solution of indicator to have the appropriate concentration to discolor when one drop is added to *V* mL of a solvent containing more than *p* ppm water.

Mass of Ph_2CO in a drop of solution: $(p \times V \times d_{solvent} \times FW_{Ph2CO}) / FW_{solvent}$
Volume of one drop (disposable Pasteur pipet) ~ 0.013 mL (153 drops for 2 mL of THF)

Mass of Ph_2CO for 20 mL indicator solution:
$(p \times V \times d_{solvent} \times FW_{Ph2CO} \times 153 \times 10) / FW_{solvent}$

For testing THF:

$FW_{solvent} = 72$ g mol^{-1}
$d_{solvent} = 0.889$ g mL^{-1}
V = 4 mL
$p = 10 \times 10^{-6} = 10$ ppm

Hence, $10 \times 10^{-6} \times 4 \times 0.889 \times 182 \times 153 \times 10 / 72 = 0.137$ g Ph_2CO for 20 mL indicator solution. Excess sodium was used (28 mg, 1.6 equiv).

Concentration of Ph_2CO in solution is 0.0377 M (moles L^{-1}).
For a different solvent, if the solution stays purple, max content of water is:
$p' = C_{Ph2CO} \times 0.000013 \times FW_{solvent} / (V \times d_{solvent})$

For pentane:
$FW_{solvent} = 72$ g mol^{-1}
$D_{solvent} = 0.63$ g mL^{-1}
$V = 4$ mL

⇨ $p' = 14$ ppm

Given that the solution prepared above precipitates a little solid, the concentration of ketyl radical is slightly lower, so the concentration of water in the solvent is even smaller. If even more rigorously dry solvents are required, the volume of solvent tested could be doubled.

C. Training

All users should receive training for each specific glovebox. A detailed training process has been developed. One main training is completed first, and then additional box-specific training is required before using any other gloveboxes.

D. Troubleshooting

Category	Problem	Signs	Solution	Notes
Noises	Box leak	Frequent refilling	Close all ports of vacuum line, check antechamber door seal, check for holes in gloves	Can also be caused by a septum on the vacuum line not sealing properly
Noises	Failed purge	Clicking sound	Change nitrogen tank	
Noises	Broken (or breaking) fan	Louder whirring	Pump new fan in overnight to replace	
Noises				
Warning	Loose electrical connection for freezer temperature sensor	Warning stating "Freezer temperature too high"	Tighten electrical connection attached to T sensor on top of glovebox	For pictures, see section VII. Freezers and Cold Wells