MBraun Glovebox

Please see Mitchell and Kendra for training before your first use.

Preparing Items To Be Brought Into the Glovebox

Equipment

Glassware, stirbars, needles, spatulas and pretty much anything that can go in the oven: should be dried in the oven for at least one hour prior to being brought into the glovebox.

Plastics, syringes and other stuff that cannot go in the oven: should be pumped down on a schlenk line, in the large anti-chamber or in the small anti-chamber (if no one needs to use the glovebox) overnight (for a lot of stuff) or an hour (for small amounts).

Commercial Chemicals

Inorganic salts: should be dried according to standard procedures, typically this entails heating under vacuum on a Schlenk line.

Non-volatile chemicals packed under argon: New chemicals that arrive packed under argon can be brought directly into the glovebox. Do not open them until they are under inert atmosphere!

Non-volatile chemicals not packed under argon: should be dried under high-vacuum by loosening the cap of vial prior to bringing them inside.

Sure-seal bottles: Chemicals in Sure-seal bottles can be brought directly into the glovebox, provided they are completely unopened (the cap has not been untwisted and/or the seal punctured). Once opened in the glovebox, these chemicals should be transferred to schlenk tubes, or stored in a secondary container. If the seal has been broken, the chemical should be transferred to a schlenk tube and sparged with nitrogen.

Volatile chemicals: Dichloromethane, toluene, tetrahydrofuran, ether and hexanes are supplied directly from the solvent system to taps within the glovebox. Other acceptable solvents such as benzene or pentanes should be dried according to standard procedures (distillation), transferred to a schlenk tube or solvent bulb and deoxygenated (sparged with nitrogen or subjected to 3 freeze-pump-thaw cycles) before bringing them into the glovebox.

Homemade Chemicals

Nonvolatile solids and liquids: should be dried under high-vacuum by loosening the cap of vial prior to bringing them inside.

Volatile solids: should be sublimed, transferred to a vial and brought in via a sealed vacuum chamber.

Volatile liquids: should be distilled into a schlenk tube.

Other Items

Kimwipes should be oven dried for 6-8 hours (be sure to cut off the plastic part of the Kimwipes package or it will melt in the oven) and then placed in the antechamber while hot and left in the chamber under vacuum overnight before being brought in.

Bringing items in the glovebox

Using the small antechamber

1. The small antechamber is generally kept under dynamic vacuum.


2. To load items ‘refill’ the anti-chamber to box pressure.


3. **Close the refill valve by turning the stopcock up**. Note: if you fail to close the valve before opening the outer door, then the interior of the box will be directly exposed to the outside atmosphere!

4. Open the antechamber door and load the required equipment into the antechamber.

5. Evacuate the antechamber.

6. After approximately 5 minutes, refill the chamber to -10 psi then immediately evacuate the chamber again.

7. Repeat step 6 two more times.

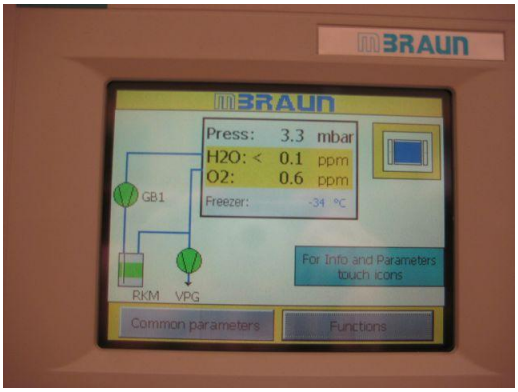
8. On last cycle, refill completely to box pressure and then close the refill valve by turning the stopcock up. Open inner door and take your things into the box, however reclose the inner down while working in the box. Evacuate the antechamber. If you are planning to use solvents in the box, make sure you read *section III* (**Using Solvents in the Glovebox)**

9. When you are done working in the box, partially refill the chamber to 10 psi by turning the stopcock to the right and close the valve. Place your items in the chamber and close the inner door. Again, make sure that the refill valve is closed before opening the outer door.

10. Close the outer door and place the chamber under dynamic vacuum when finished.

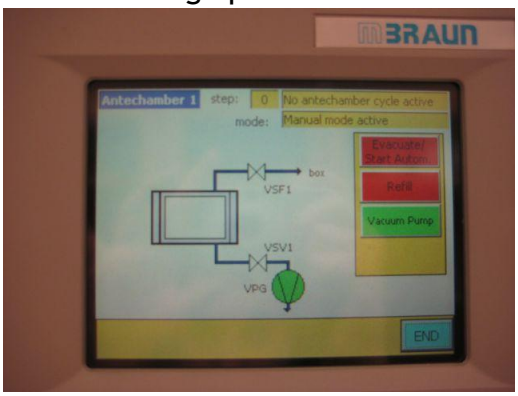
Using the large antechamber

1. The large antechamber is generally kept under static vacuum and is controlled via the touch panel.



To access the controls, tap the blue box on the main menu. Be sure that the small antechamber is set to: closed.

This will bring up the antechamber screen:



1. To refill the antechamber, tap the *REFILL* button. It will turn green and you will hear the box filling and see the pressure change on the gauge. Manually press on the gloves to limit the audible clicking.

2. When the pressure reaches box pressure, close the refill valve by tapping *REFILL* again. The button will turn red.

3. Open the anti-chamber door and load the required equipment into the anti-chamber. Close the door. (Caution: Do not over-tighten!!)

4. Evacuate the anti-chamber by tapping the *EVACUATE/START AUTOM* button. The button will turn green and the chamber will evacuate.

5. After approximately 40 minutes, tap the *EVACUATE/START AUTOM* button to close the valve (it should turn red). Then refill the chamber by tapping *REFILL* (it should turn green).

6. Repeat steps 4 and 5 two more times. Alternatively the large anti-chamber can be pumped down in a single overnight cycle.

7. On last cycle, refill completely to box pressure and then close the refill valve tapping *REFILL*. Verify that both the *REFILL* and *EVACUATE/START AUTOM* are red. Open inner door and take your things into the box, however reclose the inner down while working in the box.

8. When you are done working in the box, place your items in the chamber and close the inner door. Again, make sure that the refill valve is closed before opening the outer door.

9. Close the outer door and place the chamber under static vacuum by (i) tapping *EVACUATE/START AUTOM* (turns green) until the chamber is completely evacuated and then (ii) tapping *EVACUATE/START AUTOM* (turns red) to close the valve.

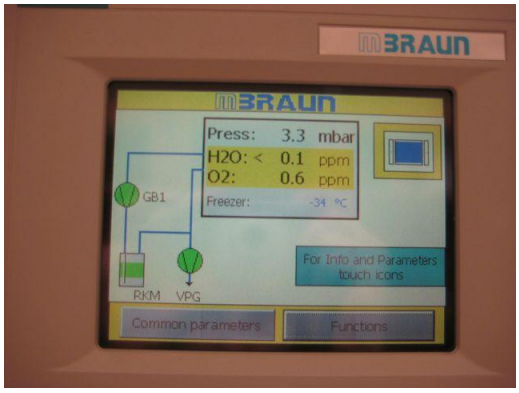
Using Solvents in the Glovebox

Caution: You should always turn of the 'CIRCULATOR' and 'ANALYZER' before using any volatile chemical or solvent in the box (see below).

This prevents solvents from getting into the circulator and destroying the catalyst that cleans the atmosphere of the box. The catalyst is a copper complex and typical reactivity trends that apply to other metal complex also apply to it. Also, remember that the box is a closed system. Therefore, any solvent that you open enters the atmosphere and will contaminate any solvent that you open subsequently. After using solvents or other volatiles the box must be purged for at least 30 minutes to flush the atmosphere.

Turning off the circulation

1. From the main menu tap the grey *FUNCTIONS* button on the lower right side of the screen. A new screen will appear.



2. Tap the green *CIRCULATION* and *ANALYZER* buttons. They will turn red and the circulation will audibly cease.

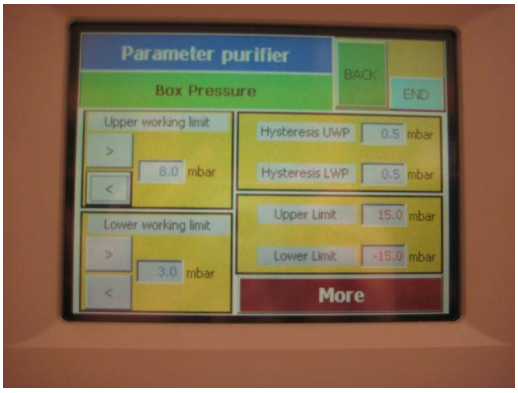
3. You can now use solvents in the box.

4. To return to the main menu tap the blue *END* button in the top left.

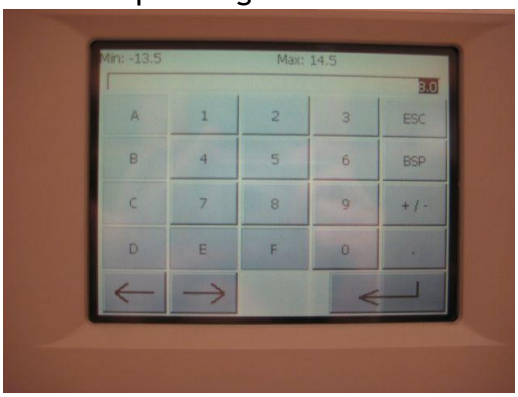
Starting the purge

1. Before purging be sure all containers containing volatiles are tightly sealed with Teflon lined caps and all pipettes, syringes, kimwipes and anything else contaminated with solvents have been removed from the box.

2. From the main menu tap the white *PRESSURE* bar. This will bring up the parameter purifier screen.



Change the upper and lower limits to 14 and 11 respectively by tapping the respective parts of the screen entering the values on the keypad that appears and then pressing enter.

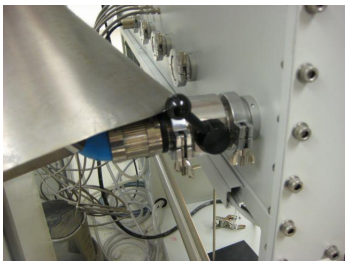


END button! Very Important!

After you press enter you will return to the parameter purifier screen.

3. After adjusting the pressure tap *END* to return to the main menu. This is crucial because the box will not adjust the pressure AT ALL if the adjustment screen is up. After taping *END* you should audibly hear the box fill and observe the gloves stiffen.

4. After the pressure in the box adjusts, open the manual purge valve to where it meets the metal exhaust cone (about half way).



5. You should hear nitrogen escaping from the box. Take a look at the monitor screen to verify that the box is holding positive pressure (around 8 psi). Do not touch the box during the purge.

Ending the purge

1. Close the manual purge valve.
2. Return the upper and lower limits to 7 and 3 respectively.
3. Turn the circulation and analyzers back on (the buttons will turn green).

Turning on and off the light

1. 1. From the main menu tap the grey *functions* button on the lower right side of the screen. A new screen will appear.
2. Tap the *box light* buttons (green when light is on, red when light is off).
3. The light should be turned off at the end of each working day.

Using the vacuum pump

1. Set up the trap, but do not cool it yet.
2. Open the valve (inside box) by pointing it toward the back of the box. Turn on the vacuum pump and then quickly close the valve by turning it perpendicular to the back of the box.

3. Now you can cool the trap and use the vacuum.
4. To shut off the vacuum: Lower the dewar, open the valve then quickly turn off the vacuum. Finally, be sure to close the valve.

Other things to know about working in the glovebox

- Please keep the box clean and organized! There is masking tape in the box for cleaning up spills of solids.
- Try to avoid spilling solvents on the gloves as it will wear them out faster
- Be cautious with sharp pointy things around the gloves (needles, copper wire, broken glass) so as you run the risk of poking a whole in one of the gloves. If you do poke a hole (or find a hole) in a glove, cover it with electrical tape on both sides and let Amanda or Mitchell know.
- Destatic things as much as possible using the static gun and keep as still as possible when using the balance in the box, as slight changes in pressure will affect the reading.
- If supplies in the box are running low let Amanda or Mitchell know so they can refill them.