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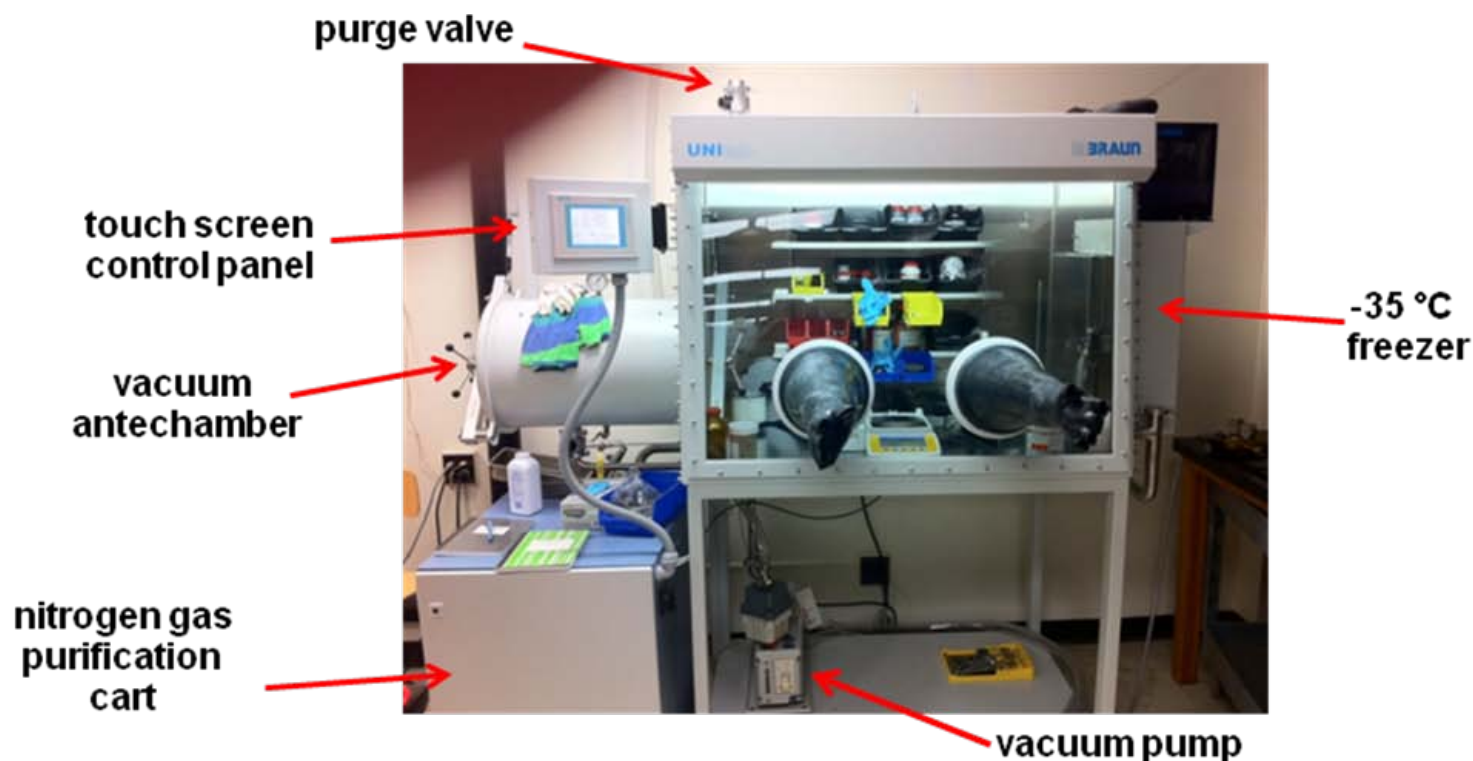
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Burns Group mBraun Glovebox Standard Operating Procedure (SOP)

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mBraun contact person: Tom Wagener, 1-603-773-9333 ext. 243

mBraun Glovebox is located in Chemistry 340.



Introduction to the mBraun glovebox

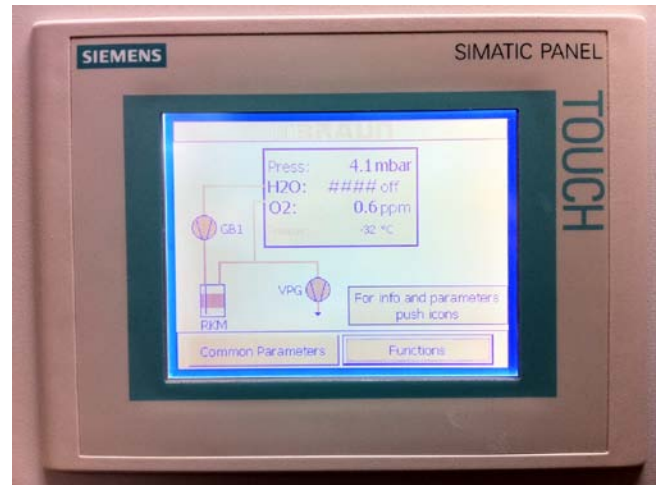
The mBraun glovebox is operated using an inert gas (N_2) enabling the user to handle substances which are sensitive to oxygen and moisture. This glovebox is operated using a closed loop circulation method to provide the user with a non-reactive N_2 atmosphere of <1 ppm moisture and oxygen. See the mBraun glovebox manual (red 3-ring binder) for a more detailed description of all the operations of the glovebox.

Before using the mBraun glovebox.....

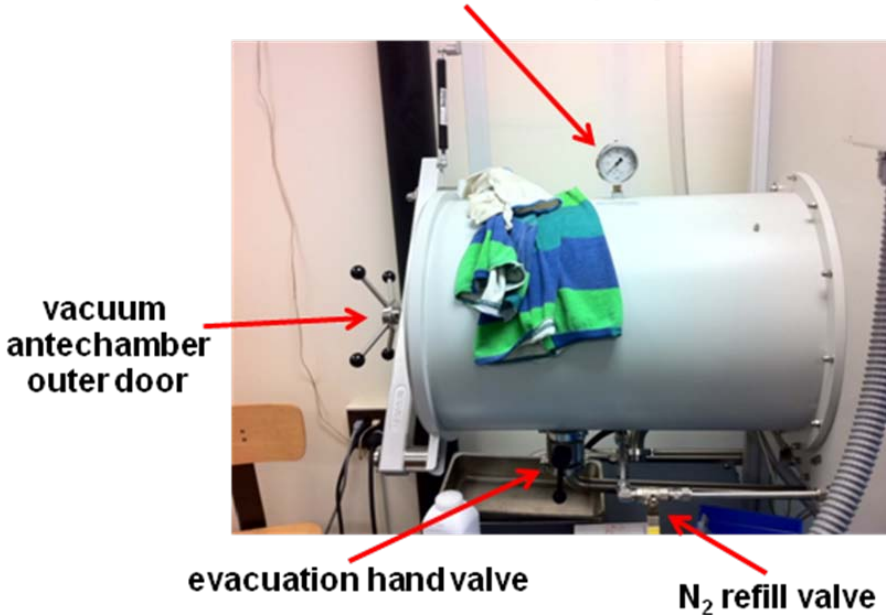
1) Before you start – Look at the touch screen control panel. Check the current N₂ pressure inside the glove box... it should be between 4 and 8 mbar. Check the O₂ level... ideally it should be less than 5 ppm. Contact Dr. Burns ASAP If either of these two conditions are not met. Note: there is not an H₂O meter on this glovebox. That is why no value is indicated for the H₂O level.

2) Check the mBraun log book sitting on top of the N₂ gas purification cart to make sure no one is currently using the glovebox and/or the vacuum antechamber to send something into the glovebox.

3) Check the antechamber vacuum gauge. When the antechamber is not being used it is to be left under dynamic vacuum so the vacuum gauge on top of the chamber should read around -30 in Hg.



antechamber vacuum gauge



antechamber vacuum gauge

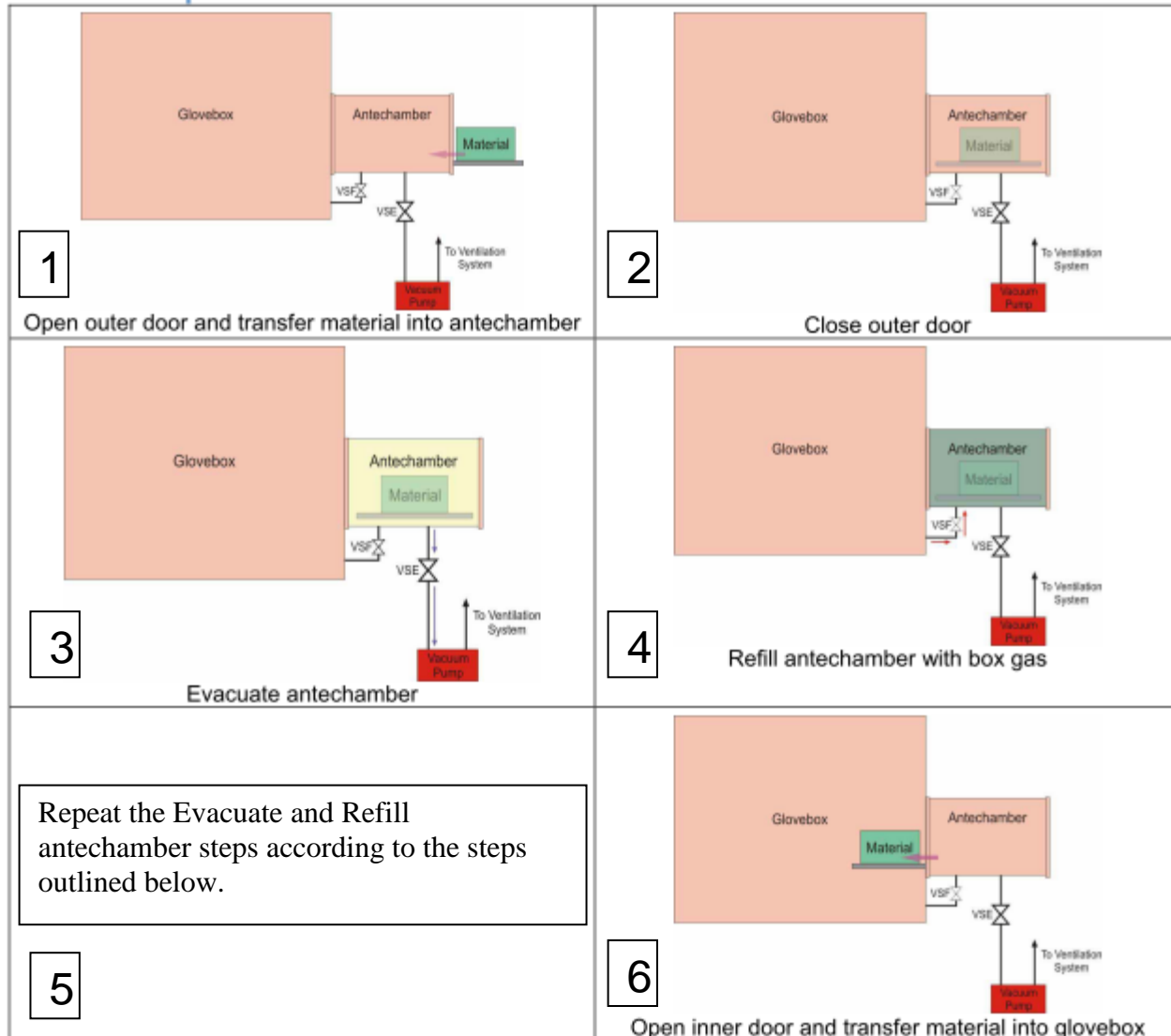
Vacuum Antechamber - General Information

The glovebox is equipped with a large antechamber designed for transferring material into or out of the glovebox without polluting the internal box atmosphere or the lab atmosphere. The antechamber is equipped with an inner door, outer door and sliding tray to aide in transferring material. Transferring material into and out of the box is a completely manual procedure which utilizes the antechamber and its related components. The chamber undergoes several evacuation/refill cycles to clear the chamber of room or glovebox atmosphere prior to removing the material from the chamber.

- Note: 1)** An evacuated chamber cannot be opened. Attempting to open an evacuated antechamber may damage the door locking mechanism.
- 2)** Keep both antechamber doors closed and under dynamic vacuum when it is not being used to transfer material in or out of the glovebox.

Below is a general description of vacuum chamber operation.

Method of Operation



Vacuum Antechamber Operation

Transferring Material into the Glovebox using the vacuum antechamber

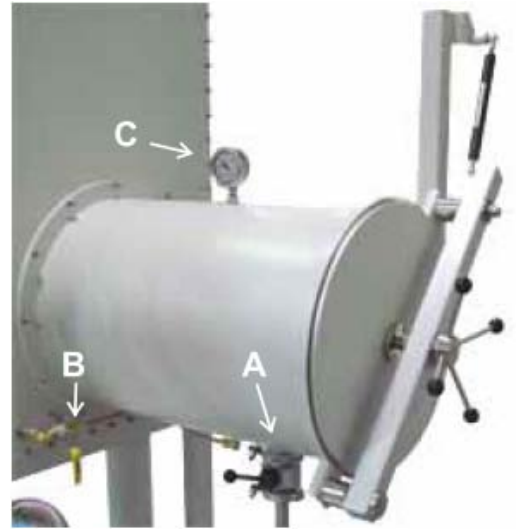
Follow the steps below to perform a transfer of materials from the lab into the glovebox using the vacuum antechamber.

1) Before opening the outside antechamber door, make sure that:

- The inner antechamber door is closed;
- The evacuate hand valve (A) is in the closed position;
- The refill hand valve (B) is in the closed position;
- The antechamber is not under vacuum (gauge (C) should read 0 in. Hg.).

The picture to the right displays the correct hand valve position and the reading on the gauge at the zero position.

NOTE: Make sure to fill out the mBraun Glovebox log book as you use/enter the antechamber. See Dr. Burns or senior lab members for instructions on how to do this.



2) Turn the handle on the outer antechamber door in a counterclockwise direction to open it.



3) Carefully open the antechamber door by lifting it in an upward direction.





4) Let go of the door handle and allow the door to rise completely.



5) Gently pull out the sliding tray.



6) Place the materials to be introduced into the glovebox (in a secondary metal spill tray whenever possible) on the sliding tray and slide the tray back into the antechamber.



7) Gently push the sliding tray back into the antechamber.

8) Close the outer door by pulling down until the groove at the end of the arm rests on the spindle.



9) Place four fingers, two each on opposite spindles, on the door handle and gently turn in a clockwise motion until the door touches the chamber. Tighten $\frac{1}{4}$ of a turn more.

10) Evacuate the antechamber by turning the evacuation hand valve to the open position.



11) Wait for the gauge to settle at the **negative** position (-30 in Hg.) and continue to evacuate for 15-20 minutes.



12) After the waiting for ~20 min. turn the evacuation valve to the closed position.



13) Refill the antechamber to -10 in. Hg. by turning the refill valve to the **open** position. N₂ gas is delivered to the antechamber from inside the glovebox. Use the glovebox foot pedals to maintain the appropriate pressure inside the glovebox (between 4 and 8 mbar).



14) Turn the refill valve to the **closed** position.



15) Repeat the process as outlined below to complete 3 total evacuation/N₂ refills of the antechamber ...

Evacuation (3 total cycles)

- 2 additional cycles – 15-20 min. each

Refill (3 total cycles – 2 intermediate, 1 final)

- 1 intermediate cycle – refill chamber to -10 in. Hg.
- 1 final cycle – refill chamber to atmospheric pressure (0 in. Hg. on the gauge)

16) Once your material in the antechamber has undergone the evacuation/refill process as described above and the antechamber is under atmospheric N₂ pressure place your hands in the gloves of the glove box. Grasp the inner door handle with two fingers and turn it in a counterclockwise motion.



17) Carefully open the antechamber door by lifting it in an upward direction and guiding it until it stops.





18) Gently pull out the tray and transfer the material into the glovebox.

At this time, materials to be transferred out of the glovebox may be placed on the tray.

Recommended: Close the inner door right after bringing in your material.

19) After all materials are transferred into the glovebox and any materials to be transferred out of the glovebox are on the tray... grasp the door handle and gently pull the door down until it rests on the door stop.



20) Grasp the door handle with two fingers and turn it in a clockwise fashion until the door touches the chamber. Tighten $\frac{1}{4}$ of a turn more.

NOTE: If manipulations in the glovebox must be performed after bringing items into the glovebox and before removing items from the glovebox... closed the inner chamber door as described above and leave the chamber under static N_2 pressure until you are ready to remove items from the glovebox.

REMEMBER: Fill out the mBraun Glovebox log book as you use/leave the glovebox. See Dr. Burns or senior lab members for instructions on how do this.

Working Inside the mBraun Glovebox

- 1) When working in the glovebox use of nitrile inner gloves is mandatory in order to protect the glovebox butyl rubber gloves.
- 2) You can adjust the lower pressure limit to either 1 or 2 mbar when working in the glovebox.
- 3) When you take more than a 5 minute break or are done working in the glovebox set the lower limit back to 4 mbar.
- 4) Use of solvents and volatile reagents inside the mBraun glovebox is prohibited. If you feel the use of such a reagent is necessary discuss this with Dr. Burns first. Dr. Burns' permission is required before any such manipulations can be carried out inside the mBraun glovebox.

Useful Glovebox Materials

- 1) The person in charge of the glovebox will try to maintain a common supply of gloves, weighing boats, weigh paper, Kim wipes, grease etc..
- 2) If you find it necessary to add any of these materials yourself, please watch for the following points.
 - Any kind of glassware, also items such as spatulas, should be heated in the oven (minimum 120 °C) for a minimum of 2 hours, preferably overnight.
 - If for whatever reason you need to bring into the glovebox glassware that wasn't heated, it is recommended to evacuate it overnight before glovebox entry.
 - Weighing boats can enter the box by normal pump down procedure and do not require drying.
 - Kimwipes are dried in the vacuum oven (see vacuum oven SOP for details) and evacuated overnight in the glovebox antechamber before entry into the glovebox.

Precautionary Items

- 1) The upper pressure limit on the control panel must be maintained at 8 mbar.
- 2) Whenever working with any reagents that can contaminate the atmosphere or damage the catalyst (Dr. Burns' permission required prior to use), turn off the O₂ analyzer, circulation, then purge the glovebox (about ~ 100 psi N₂ gas – 10 to 20 minutes), and turn on the circulation again. After 30 minutes with the circulation running you can turn on the O₂ analyzer. Purging the mBraun glovebox is described below.
- 3) If the pump must be turned off (oil change, etc.) turn off the O₂ analyzer and circulation first.
- 4) Only the person who is responsible for the glovebox conducts regenerations of the catalyst (with a mixture of 5% H₂ and N₂ as the carrier gas).

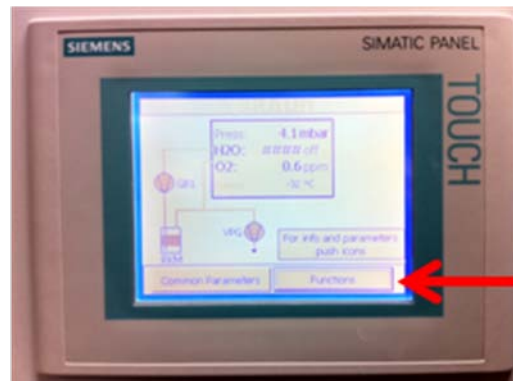
Housecleaning and Storage of Chemicals

- 1) All containers and wessels containing chemicals must be clearly labeled and placed in their appropriate place (i.e. no random storage of materials on the floor of the box).
- 2) Take out your waste on your own.
- 3) Use the secondary metal tray used to sent your item into the glovebox to contain any spills you might have while working in the glovebox. If you spill something on the floor of the box or on the balance use the hand vacuum inside the glovebox to clean it up.
- 4) If you use something, put it back. If you exhaust something, replace it or notify the person in charge of replacing it.
- 5) If you must bring a metal item (spatula, forceps) outside the glovebox to clean it... once clean place it in the oven for a minimum of two hours to dry it and return it to the glovebox hot (see above).

Purging the mBraun Glovebox

- 1) To purge the mBraun glovebox you must first turn off the O₂ analyzer, Circulation, and Freezer.

- On the “start screen” of the touch screen control panel touch the “functions” key in the lower right corner of the display.

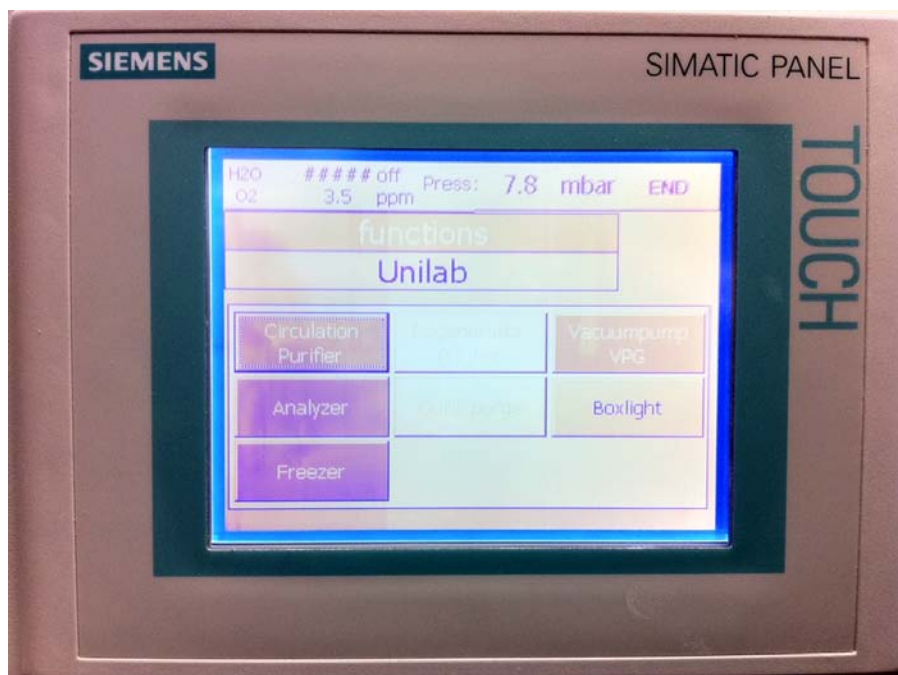


Functions Key

- This takes you to the “functions” screen. When a component of the mBraun glovebox is active (on) it's corresponding “function key” will be darkened.

In the photo to the right the...

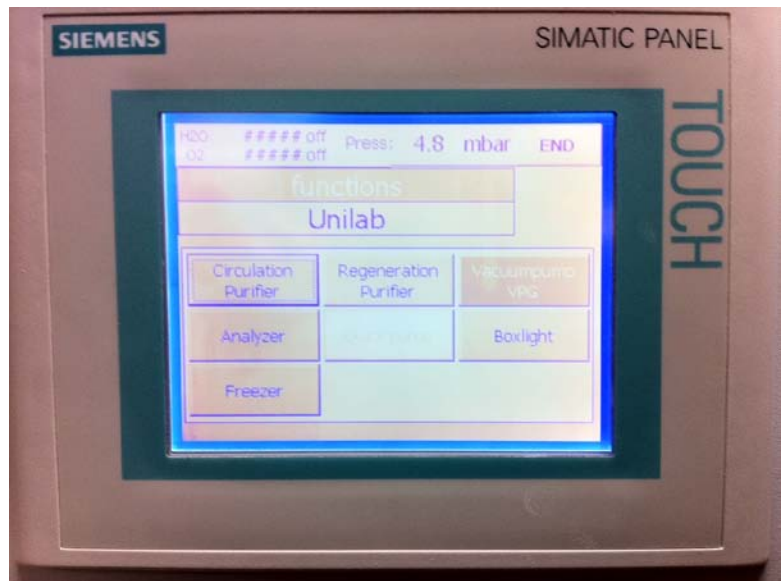
- 1) Circulator is active
- 2) Vacuum pump is active
- 3) O₂ analyzer is active
- 4) Freezer is active
- 5) Glovebox light is **not active** (unlit function key)



- Touch the Circulator, O₂ analyzer, and freezer buttons to turn them off and the screen will look like picture shown.
Note: Do not turn off the vacuum pump.

In the photo to the right the...

- 1) Circulator is off
- 2) **Vacuum pump is on**
- 3) O₂ analyzer is off
- 4) Freezer is off
- 5) Glovebox light is off



- 2) Open the freezer door inside the glovebox. This is done so any impurities possibly trapped in the freezer will be removed from the glovebox when the atmosphere is purged.

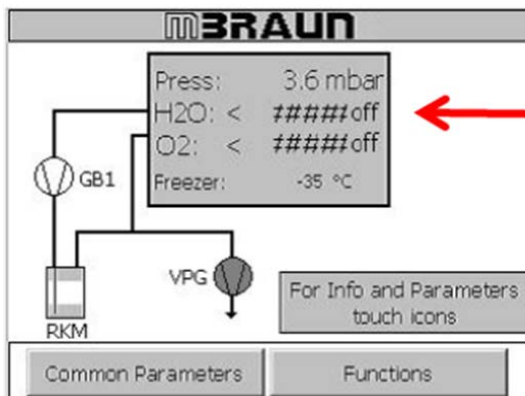


Freezer Door Closed



Freezer Door Open

- 3) Touch the **Box Pressure/Analyzer** display box on the start screen.



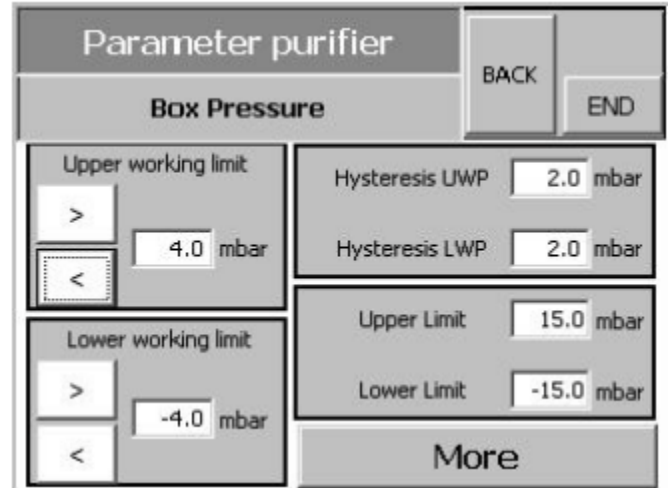
Box Pressure/Analyzer display box

4) There are two ways to adjust the internal N₂ pressure of the glovebox...

- a) Use the arrows listed under Upper Working Limit and Lower Working Limit to increase or decrease the box atmosphere pressure set point in one tenth decimal intervals

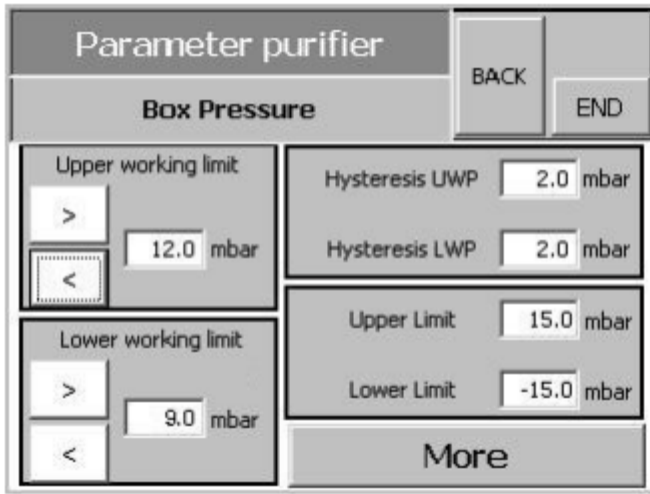
OR

- b) Touch the number field next to the corresponding parameter to display the alphanumeric keypad. Use the keypad to adjust the parameters in the same manner as before.



Setpoints for Purging the mBraun Glovebox

- Upper Working Limit: 12.0 mbar
- Lower Working Limit: 9.0 mbar



5) Touch the End Button.

6) Open the manual hand valve located on the top of the glove box.



Closed



Open

The VG valve will open and close to allow working gas (N₂) to flow into the glovebox. Once the pressure inside the glovebox drops to a level between 1.0 mbar and 8.0 mbar, the valve will stay open (no clicking noise is heard) providing a constant flow of gas into and out of the glovebox. Purge for a minimum of 10 minutes and a maximum of 20 minutes.

7) Once N₂ purge is complete, close the manual hand valve and reset the internal glovebox N₂ pressure to between 4 mbar and 8 mbar.

8) Close the freezer door, turn the freezer on, and turn the circulator on. After the circulator has run for 30 minutes turn on the O₂ analyzer and check the O₂ level in the glovebox.