U N I V E R S I T Y *of* **H O U S T O N**
Miljanić Research Group ▪ Department of Chemistry ▪ 136 Fleming Building ▪ Houston, TX 77204-5003
nsm.uh.edu/~miljanic/ ▪ miljanic@uh.edu ▪ 713.743.2718 (office) ▪ 713.743.2717 (lab)

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Glovebox Use Guidelines

Group contact person: **Karolina Osowska** in 204 Fleming, email: karolina.osowska@gmail.com

Click here for the full version of VAC Atmospheres glovebox manual.

VAC Atmospheres glovebox is located in 204 Fleming. The group inherited this glovebox from the late Prof. Jay K. Kochi. It was not operational when the group received it, and was extensively inspected and repaired by Ognjen before being put into use. The repairs included: (1) inspections of solenoid valves; (2) replacement of all hose connections for gases; (3) installation of new water filter and two new air filters; (4) replacement of the compressor for the freezer; (5) replacement of copper tubing for inert and regeneration gases; (6) fabrication of shelves for the freezer; (7) replacement of the vacuum pump and vacuum tubing; (8) addition of a purge valve on the backside of the glovebox, and (9) thorough cleaning. After three regeneration cycles, the glovebox was deemed operational.

Introduction to the Glovebox

Glovebox is an instrument that allows convenient manipulations of materials in an atmosphere of an inert gas, most commonly nitrogen. The glovebox atmosphere is continuously circulated over a bed of **catalyst** which removes traces of water and oxygen. The catalyst is a combination of copper-based particles (that remove oxygen down to 1 ppm) and molecular sieves (that remove water down to 5 ppm). The catalyst needs to be regenerated periodically; instructions on how to do that are given below.

The glovebox is generally used for the manipulation of air- and water-sensitive materials. Manipulations inside the glovebox differ very little from their counterparts outside of it: handling of materials is done using thick butyl **gloves** and the common operations of stirring, weighing, and refrigerating materials can be easily reproduced. These gloves are the weakest point of the glovebox and its most common source of contamination, since it is very easy to introduce air into the glovebox through small incisions and holes in the gloves. These incisions can be made while handling sharp objects, like spatulas, syringe needles, scissors, etc. The gloves are much thicker than normal nitrile (or latex) gloves, so they will take some getting used to.

In order to be able to work in the glovebox, you will need to bring materials and equipment into the glovebox without compromising its atmosphere. Your only "gate" to the glovebox is the **antechamber**—large cylindrical assembly on the left-hand side of the box. It has two doors—one that can be opened only from the inside of the box and one that can be opened only from the outside. These two doors should NEVER be open at the same time. It also has two valves on it—one connecting it to the vacuum pump (antechamber vacuum valve), and another connecting it to the glovebox (this valve is the source of inert gas for the antechamber). These two valves should also NEVER be open at the same time.

The pressure of the inert gas within the glovebox should always be kept slightly above atmospheric pressure, to avoid contamination by back-sucking air into the glovebox. The glovebox regulates its pressure automatically. For quick manual adjustments of glovebox pressure, you will often use its **pedatrol**—a foot pedal which is lying on the floor and which allows quick control over the glovebox pressure. If you see that the pressure is too high (it is easy to feel, gloves "oppose" you) press the left pedatrol button to decrease it ("low"). If the pressure is too low (gloves are hanging down or are sucked inside the box) then press the right button to increase it. Obviously, do not press both buttons at the same time.

Using the Glovebox

General Guidelines

1. The most obvious thing: NEVER put anything containing oxygen (closed vial would be a good example) or water/moisture into the glovebox.
2. ALWAYS record all significant events—catalyst regeneration, nitrogen tank exchange, any malfunctions, changes of pump oil, changes of filters, replacements of glovebox parts—in the provided logbook, along with your name and the date of the event.
3. NEVER leave both the inner and the outer door to the antechamber open.
4. <u>When in doubt—pump it out!</u> If you are not sure whether your antechamber is under nitrogen or vacuum, ALWAYS pump it out. If you are not using it, leave the antechamber under vacuum.
5. When working with substances with very fine particle sizes, and you are afraid they could spill all over the place, turn off the **CIRCULATOR SWITCH** during your manipulation. Make sure to turn it back on once you are done.
6. There is very little space available in the glovebox, and it must be used effectively. Common chemicals are organized into three large boxes, with (a) main group metals, (b) transition metals, and (c) organic compounds. Each user should organize their own smaller container to keep their samples in it. NEVER borrow things without asking.
7. Before putting your hands into the gloves, ALWAYS remove your watch, rings, and bracelets. This will minimize the risk of making holes in the gloves.
8. ALWAYS put on new clean nitrile or latex gloves before you enter the glovebox gloves. The

concern is purely hygienic. The atmosphere within the glovebox is about 3–7 °C warmer than the outside (because of the circulator's hot motor), so you will sweat. Use some baby powder (provided on top of the glovebox) to minimize this discomfort.

9. ALWAYS be mindful of the pressure. This is especially important if you are operating the antechamber or putting your hands in or out of the box. The pressure should not be too low (gloves sucked into the box), or too high (gloves sticking straight up from the box). Use pedatrol to adjust the pressure differences quickly; automatic pressure control will eventually kick in. If it does not, something is wrong: contact Karolina or Ognjen immediately.
10. All vessels brought into the glovebox must be nitrogen-filled or evacuated before. If you do not do this, you will introduce air into the box. Make sure that all empty containers are open. When putting in a vessel with a solvent—especially a volatile one, like Et_2O—or a solution, the stopper must be properly secured, even on flasks which were evacuated before. If not, it will very likely pop out and make a big mess in the antechamber. Use plastic clips and additional rubber bands to hold the stopper in place.
11. All solvents or solutions that you plan to put into the box must be dry and degassed.
12. The glovebox catalyst can be irreversibly damaged by thiols, amines, phosphines, alkyl halides, SO_2, SO_3, Hg, etc. You should avoid these materials in the glovebox at all cost. If you absolutely have to use them, ALWAYS discuss your experiment in detail with Ognjen before trying something potentially harmful.
13. In the glovebox, keep your samples open only when necessary. Do not leave any open vials in the box after you are done, since they will be thrown away.
14. When weighing materials in the glovebox, static electricity is a big problem. To minimize this disturbance, handle all materials that you plan to weigh using the provided tongs—not your gloves directly! Use the provided antistatic gun to "shoot" at the balance, tongs, and all of your vials/vessels before you place them on the balance.
15. When you are done using the box for the day, ALWAYS clean the box up! Leave it in good condition, remove all your waste. The accumulated mess in the glovebox is really hard to clean.

Bringing Materials into the Glovebox

1. Make sure that the antechamber inner door is closed.
2. If the antechamber is evacuated, close the valve to the pump.
3. Open the nitrogen valve leading to the box slowly to fill the antechamber with nitrogen. Once this is done (observe the pressure gauge), close the valve again.
4. Open the outer door, place your materials onto the antechamber shelf, and close the outer door again.
5. SLOWLY open the antechamber vacuum pump valve.

6. Place the antechamber under vacuum and keep it under vacuum for at least 20 minutes. If you are bringing in a lot of material, extend this time to 30 minutes. If you are bringing in very porous materials—paper, cork rings, kimwipes, etc.—you first evacuation cycle must last overnight.
7. Close the antechamber pump valve and slowly fill it with nitrogen again.
8. Repeat steps 5–7 two more times.
9. Put your hands into the gloves, open carefully the inner door, and bring in your materials.
10. Close the antechamber inner door.

Bringing Materials out of the Glovebox

1. Make sure that the antechamber is under nitrogen. If you are not sure about this, you must evacuate the antechamber and then refill it with nitrogen. Do not make any assumptions!
2. Open the inner door, put your stuff in, and close the inner door tightly.
3. Make sure that the valve leading from the antechamber to the glovebox is closed. Make sure that the antechamber vacuum valve is closed too.
4. Open the outer door, remove your stuff, and close the door again.
5. Slowly open the antechamber vacuum valve. Leave the antechamber under vacuum.

Catalyst Regeneration and Atmosphere Testing

The inert atmosphere in glovebox is purified by circulating it through a catalyst bed (also known as DRI-TRAIN). The catalyst bed contains a copper-based oxygen-reducing agent and molecular sieves to remove water. The catalyst bed needs to be regenerated periodically, depending on the usage and on the types of materials that were used in the glovebox. This period is typically on the order of 3–6 months. Catalyst regeneration is performed by: (a) isolating the catalyst from the glovebox, (b) exposing the catalyst to a stream of hydrogen and heating it, (c) evacuation of the catalyst, and (d) refilling of the catalyst with inert gas and reconnecting it to the box. The mixture of 5% H_2 in N_2 (uncertified) is used as a regeneration gas. One full tank is roughly enough for two regeneration cycles. During the regeneration, the chemically bound oxygen is released as water and expelled from the catalyst bed—together with the moisture released from the molecular sieves—through the stream of the regeneration gas. The full regeneration cycle takes about 12 hours: it should be started early in the morning and closely followed until the evening. Do not perform the regeneration process overnight!

Catalyst Regeneration Procedure

1. Switch the circulator off by flipping the **CIRCULATOR SWITCH** on the glovebox DRI-TRAIN control panel (see Figure). You will hear the circulator motor go quiet.
2. Isolate the catalyst from the box by turning two valves over the catalyst to horizontal position. Valves are located on the left from the DRI-TRAIN control panel, over the catalyst

bed. You will need a wrench for this purpose. It is crucial that you make sure these valves are exactly horizontal.

3. Switch the button from **CIRCULATION** to **REGENERATION** position.
4. Turn the **REGENERATION TIMER** clockwise to **PURGE** position. Open the tank with the regeneration gas, and adjust the gas flow to about 60 cf/h, as measured by the flow meter. Never turn the **REGENERATION TIMER** counterclockwise, since this might seriously damage it.
5. Turn the **REGENERATION TIMER** clockwise to the **END** position.

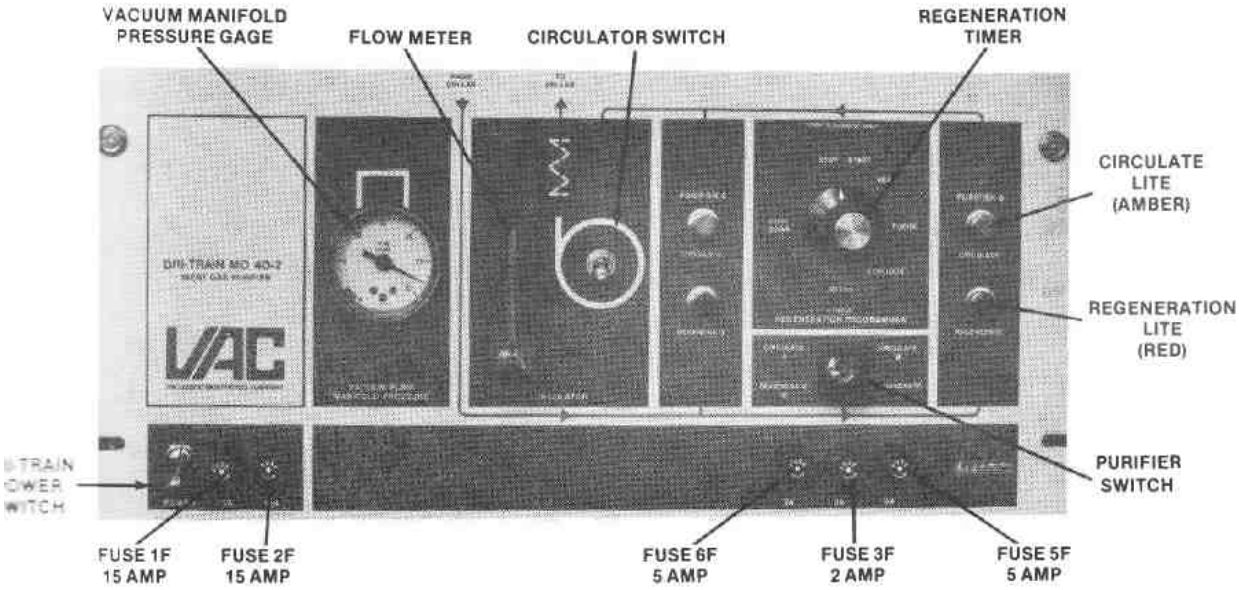

Figure. DRI-TRAIN control panel.

6. Turn the **REGENERATION TIMER** clockwise to **START**. The red light should switch on. The regeneration process will now be performed under the automatic control of the timer.
7. During the **HEAT** phase, catalyst bed will get warm to the touch. This is normal.
8. During the **PURGE** phase, water will be expelled from the catalyst. A hose is leading this water into the hood, where you should collect it in an Erlenmayer flask. Note the amount of water released. It should be on the order of 50–100 mL. If it is more, then your catalyst was really dirty, and you should run your next regeneration sooner. If it is much less than 50 mL, then either (a) you ran your regeneration too soon, or (b) your catalyst lost all of its regeneration capacity. The latter is really BAD—talk to Ognjen about remedies.
9. When the whole cycle—heating, purging, evacuation, cooling—is finished, the **REGENERATION TIMER** is again in the **END** position.
10. Switch back from **REGENERATION** to **CIRCULATION** position.
11. Turn both valves close to the catalyst to exact vertical position.
12. Turn on the circulator by using the **CIRCULATOR SWITCH**.

13. **Change the pump oil.** During the **EVACUATE** stage, much of the remaining water goes through the vacuum pump. Catalyst regeneration should ALWAYS be followed by changing the pump oil. Close the antechamber vacuum valve. Turn off the pump, then open the antechamber vacuum valve for 30 s, then close it again. Drain the oil and dispose it properly. Refill the pump with about 100 mL of fresh oil and start it. Let it run for 5 min, then turn it off again. Again, open the antechamber vacuum valve for 30 s, then close it. Drain the oil again, close the drain valve and refill the pump with oil until you reach the indicated oil level.
14. Let the glovebox run for about 24 h. After that time, you are ready to test the atmosphere within the glovebox. To perform this test, turn on a fresh 20 W light bulb that has a hole in its glass cover. The combined amount of oxygen and water will be inversely proportional to the time it takes for the lightbulb to burn out. The lightbulb should last at least several hours.
15. In the glovebox logbook, make a note of the regeneration, amount of regeneration gas used, and the volume of water released from the catalyst. Record the pump oil change.