

## ***Operating Procedure for Dry (glove) Box***

12/20/2005 Achary and Doug

This is a DRY (GLOVE) box. Therefore, NO OXYGEN AND MOISTURE exists inside of this box.

Before using the dry box, lay out a full plan of what you want to do inside of the box and make sure all the necessary items are ready with you. It is always better to put all of them together in order to avoid any additional opening of the box. This will cost the gas and time of the dry box. Before opening of the antichamber's door, make sure that nothing is inside it. A red magnet on the door indicates someone's stuffs are inside of it. In that case, consult with the earlier user first. Also, check the logbook for the information about the prior usage.

### ***Points to remember***

- \* Do not take any liquid, wet or oily materials into the dry box.
- \* All the metal tools and glassware should be heated in the drying oven prior to enter the antichamber in order to remove all the adsorbed moisture. Put them in antichamber when they are still hot
- \* Try not to bring any type of paper, rubber, wood and textile. If necessary, take them into the antichamber through overnight. For cleaning purposes, only Kimwipes (no paper towels!) is allowed.
- \* Do not take any powder materials in a fully open container, as they may fly and contaminate the dry box.
- \* Sharp objects should be avoided as much as possible. These may punctuate the gloves.
- \* When bringing bottled chemicals, first loosen the cap and remove the tape. Then, evacuate them in the antichamber. As soon as they are in the dry box, tighten the cap.
- \* Any containers that sealed under a pure nitrogen or argon atmosphere can be put inside without opening them.
- \* Any flyable light materials should be clipped together, so that they will not fly into the vacuum pump.
- \* Do not leave the antichamber's door open for long time.

Step 1. Sign on the logbook. Make sure that the inside door of the antichamber is locked properly. Close the EVAC valve (by rotating the knob to close direction). While monitoring the pressure of the dry box (photohelic display), slowly open the REFILL valve to fill up the anti-chamber. Adjust the box pressure by pressing the R side (R and L marked for raise and lower the gas pressure inside the glove box) of the pedal switch. Once the pressure of the antichamber (dial pressure gauge) equals atmospheric pressure, close the REFILL valve. Now, you can open the outside door of the antichamber. The outside door is press locked. First, loosen the door by moving the knob outward, and then open the door by rotating it up. Place all the stuffs you want to take in on the metal plate.

Once all the materials are inside the antichamber, rotate the door downward slowly, and fit to the sleeve meant for the hand. Adjust the door to match the arrow mark written on the both in anti-chamber body and door. This will provide the best fit and lock. Now lock the door by rotating the knob parallel to the anti-chamber chamber. Check that it is properly locked before going to Step 2.

Step 2. Start evacuating the antichamber slowly rotating the EVAC valve to downwards. This should be done in stepwise, like open a little bit and wait until the rotary vacuum pump stabilizes. Depending on the condition, it will take about 5 to 10 minutes to completely open the EVAC valve. Then, wait till the pressure gauge reading drops down to  $\sim 10$  mTorr. In most cases, it will take about one hour.

To knock out any adsorbent moisture, it is recommended to flush the antichamber with inert gas a couple of times. This is to be done by closing the EVAC valve and then slowly opening the REFILL valve. You will need to raise the box pressure (By R pad switch) in order to maintain the required level of the pressure. After filling the antichamber, close the REFILL valve and evacuate it slowly as directed before.

Step 3. After the complete evacuation of the antichamber, close the EVAC valve and slowly open the REFILL valve to fill the antichamber. Again, you will need to increase the pressure of the dry box by pressing the R pedal switch. Then, close the REFILL valve. Turn on the fluorescent light on top of the glove box.

***Now you are ready to work inside the dry box***

Step 4. You must remove all jewelries and watches from your fingers, hands and wrists. Also, long or sharp fingernails must be avoided. These things are likely to punch holes in gloves. You can add soft talc powders on the hands of the glove for your comfort. Wear soft cotton gloves before inserting your hands into the gloves. Insert your both hands into the gloves slowly and monitor the box pressure. Adjust the box pressure if necessary. After your hands fit completely into the gloves, wear another pair of green colored Sol-

vex gloves on the gloves inside the box. This will help to avoid contamination of the gloves from the chemicals.

Step 5. Now you can open the inside door of the antichamber. For this, slowly rotate the knob parallel to the anti chamber. This will loosen the door. Open the door carefully by rotating it upward and gently place in rest position. Take all the stuffs into the dry box and do what you intend.

***Points to remember***

\*It is better to work on a metal plate in order to prevent any chemical spillage on the floor.

\*Do not hammer or jerk directly on the floor of the dry box.

\*Do not accumulate undue stuffs inside the dry box.

\*Try not to shatter stuffs all around. Put all the waste materials in a tray and bring them out when you are done.

Step 6. Put all your stuffs in the anti chamber and close the inside door of the antichamber by rotating the door downward and putting in the proper rest position. Adjust the door plate to match the arrow marks provided on the plate and antichamber wall for the best fit. Lock the door by pressing the knob against the antichamber. Make sure that you have locked properly the door. Now take out the Sol-vex gloves and keep in a proper place. Then remove your hands slowly from the gloves. Here, you will need to increase the box pressure slowly. The pressure inside the box should be maintained such that the indicator needle should be at the mid point between two red arrows. Turn off the light of the glove box.

Step 7. Put spill over chemicals and kimwipes contaminated with toxic and air sensitive chemicals inside the metal container provided inside the dry box. Do not mix broken glasswares with the kimwipes and keep separately in the broken glass container.

Step 8. Open the outer door of the antichamber slowly and take out you stuff. Clean the antichamber plate and close it properly. Then evacuate it slowly.

Please note the exit time in the logbook.

***Use of balance inside the dry box.***

1. Turn on the balance and wait till it stabilizes.
2. Tare it
3. Use a weighing boat (mostly aluminum one) for weighing.
4. Open the balance door and put the materials for weighing.
5. Add small amounts in each time to avoid difficulty in removal as that may spill over the balance.
6. Close the balance door and note the reading.

7. Do not make fast movement of hands or gas inside the glove box. This will create air fluctuation and affect the balance reading. Do all the weighing slowly without disturbing the box pressure.
8. Take out materials from balance in a slow and gentle manner.
9. Turn off the balance after the weighing is over.

## ***Procedure for the regeneration of the catalysts (DRI-TRAIN HE 493)***

A regular regeneration of the catalyst is required for high pure gas supply inside of the dry box. The regeneration of the DRI-TRAIN HE 493 is almost automated and it needs just to observe the schedule for turning on/off and opening/closing the valves.

Fully regenerated catalysts can remove up to 5 liters of oxygen. The quality of the atmosphere is regularly checked with the bulb test and recoded in the log book.

### **VALVES**

A	Gas Inlet valve to the dry box
B	Gas Outlet valve to the dry box
E	Vent valve from the DRI-TRAIN

### **Accessories**

4 % H<sub>2</sub> +96 % N<sub>2</sub> mixture forming gas cylinder

**Step-1.**                    -Close valve A and B by turning the lever to the close direction.  
                                  -Open the Vent valve E

(This will isolate the dry box from the DRI-TRAIN)

**Step-2. (3-4 hrs)**        -Turn on the heater by pressing ON button.  
                                  -Set the TIMER to START position by rotating the knob, in the indicated direction  
                                  -Wait at least for three hours.

(The indication bulb will turn on. The timer will control the all the procedures for the regeneration. The timer will reach to the purge region after completing the heating schedule)

**Step-3. (1 hr)**            -Open the gas tank to purge out the DRI-TRAIN.  
                                  -Check the pressure level of the gas tank and make sure that it would not get used up during purge process.

(The gas tank can be opened from the beginning also as the purge and evacuate solenoids valves are automated with the timer. One full tank should last about ten times of regeneration process (pressure drop per process should be about 200 psi). Regulate the flow rate to be ~ 500 ml/min.)

**Step-4. (8-10 hrs)**      -Close the gas tank.  
                                  -Evacuation of DRI-TRAIN (automated)

**Step -5. (5 hrs)**         -Close valve E.  
                                  -Turn off the regeneration switch and wait at least 5 hours until

the heater gets cool down.

(Timer will be at the vacuum position)

**Step -6. (10hrs.)**      Open valve B and wait for at least 10 hrs.

**Step-7.**                    Open the valve A.