SOP

Glove Box-1 with Spincoater and Hot Plate

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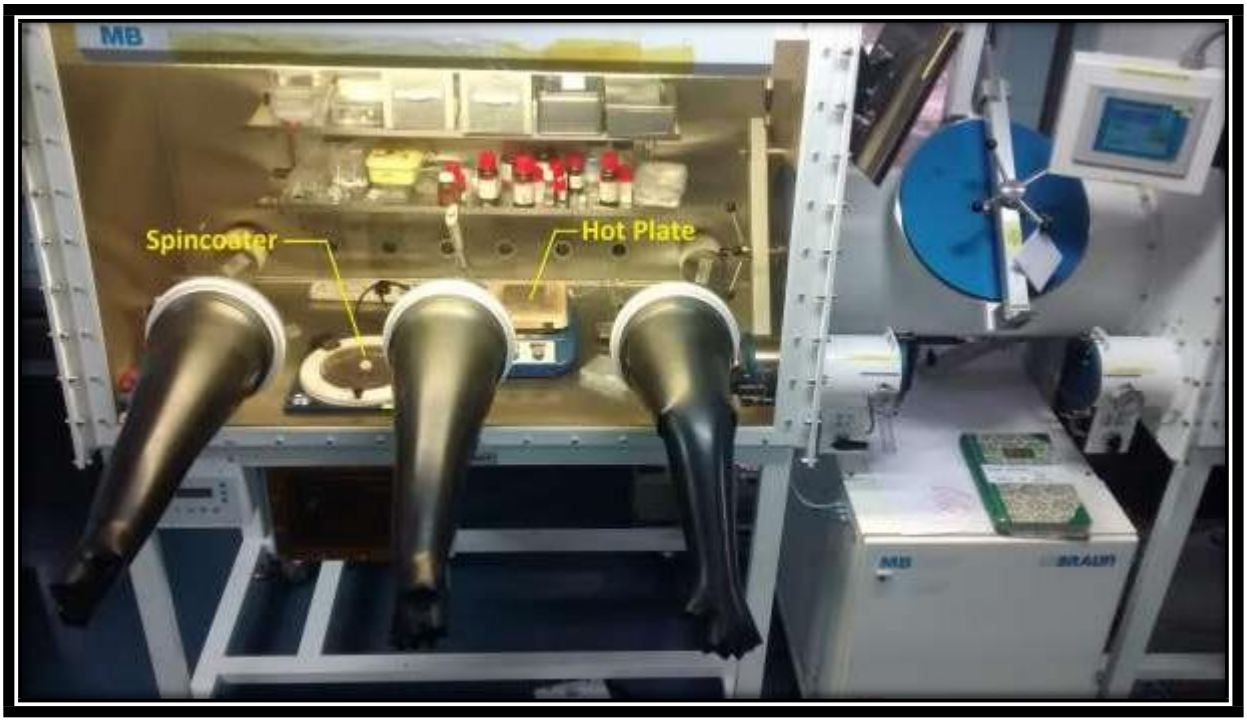
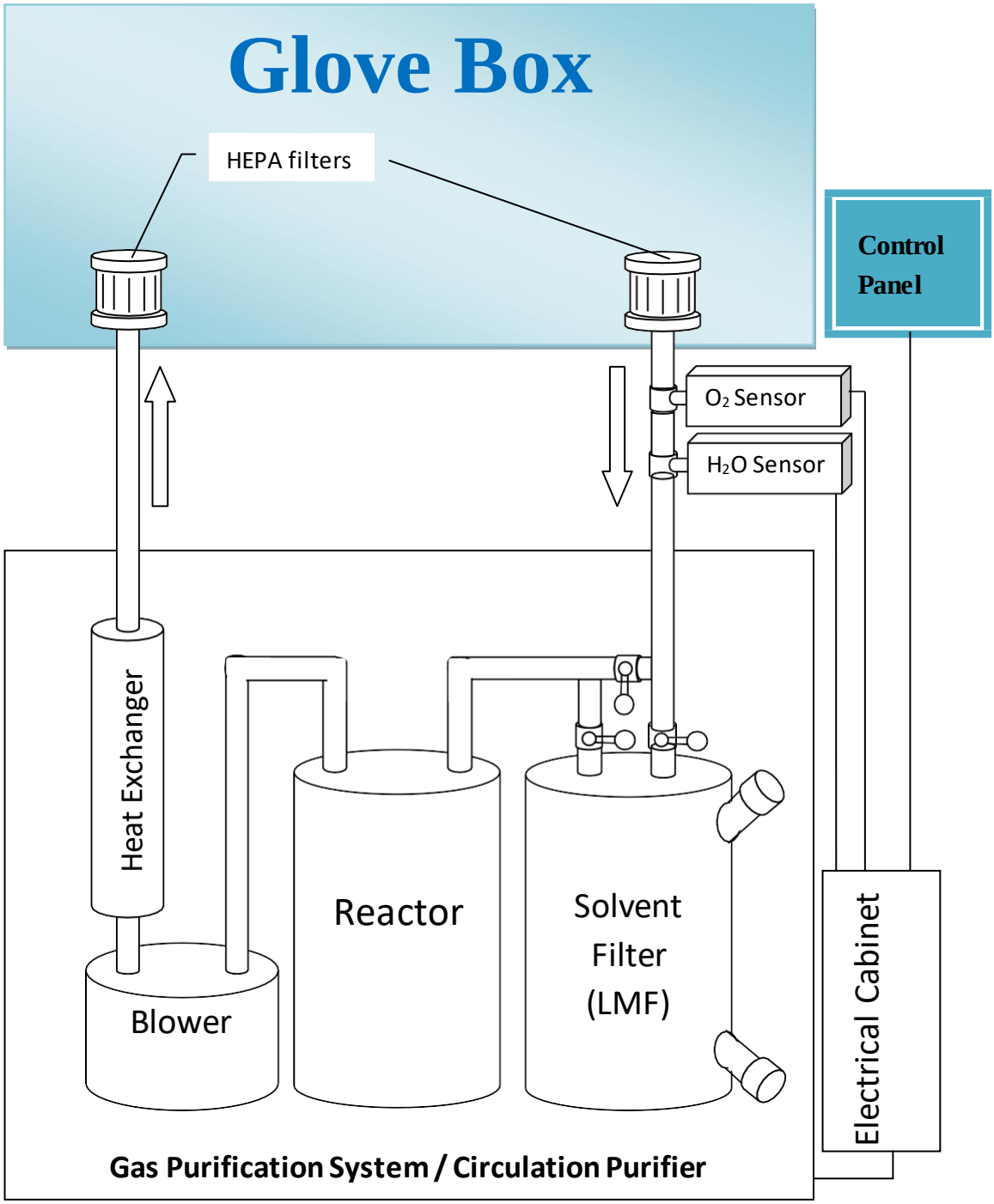


Introduction:

Generally Glove Boxes are made for conducting experiments in non-reacting ambient. Experiments which has some processing difficulty in open atmosphere due to different elements of the atmosphere are suitable to do in Glove Box. **Glove Box-1 with Spincoater and Hotplate** has *N_2 ambient* inside the box for Spincoating of solution processible organic (or H_2O and O_2 sensitive) materials and to anneal them.

A schematic diagram of GB-1 is shown as following.



General instructions:

1. After entering the Organic Device Lab first wear the clean lab shoes, lab coat, hair net, face mask and gloves.
2. Make sure to check the previous entry in the GB-1 logbook before starting any process.
3. Check N_2 gas cylinder pressure. If it is less than 10 kg/cm^2 do not start any process inside GB & contact authorized lab users to change the cylinder.
4. Make sure the line pressure is grater or equal to 6 Kg/cm2.
5. Check whether the chiller is ON and is at 16 ºC.
6. No aqueous solution is allowed inside the glovebox.
7. No sharp objects are allowed inside the glove box, because it may puncture the gloves.

Note: Only syringes are allowed for taking solvent from air tight solvent bottle inside GB-1 with a *high caution* and *covering the black gloves with* blue ones.

8. The box is controlled by PLC with Touch Panel and in Touch Panel, **Circulation Purifier**, **Analyzer** and **Vacuum Pump** should permanently be switched ON.
9. Samples Processed outside the GB should be taken inside through small antechamber with 4 times evacuation and refill cycle.
10. Make sure all the things needed for the process are there in the GB-1. Such things are:
    i. Pipet and Pipet tips of the size you require
    ii. Tweezer
    iii. Blue Extra Large Gloves

    If something is missing ask lab in-charge

NOTE: Always try to arrange the things you want to enter in the GB-1 first and then enter all things together through the antechamber to minimize gas wastage and also to reduce the chance of increase in O_2 and H_2O level. Always *evacuate for 15 min for entering gloves and lint-free cloth in GB-1.*

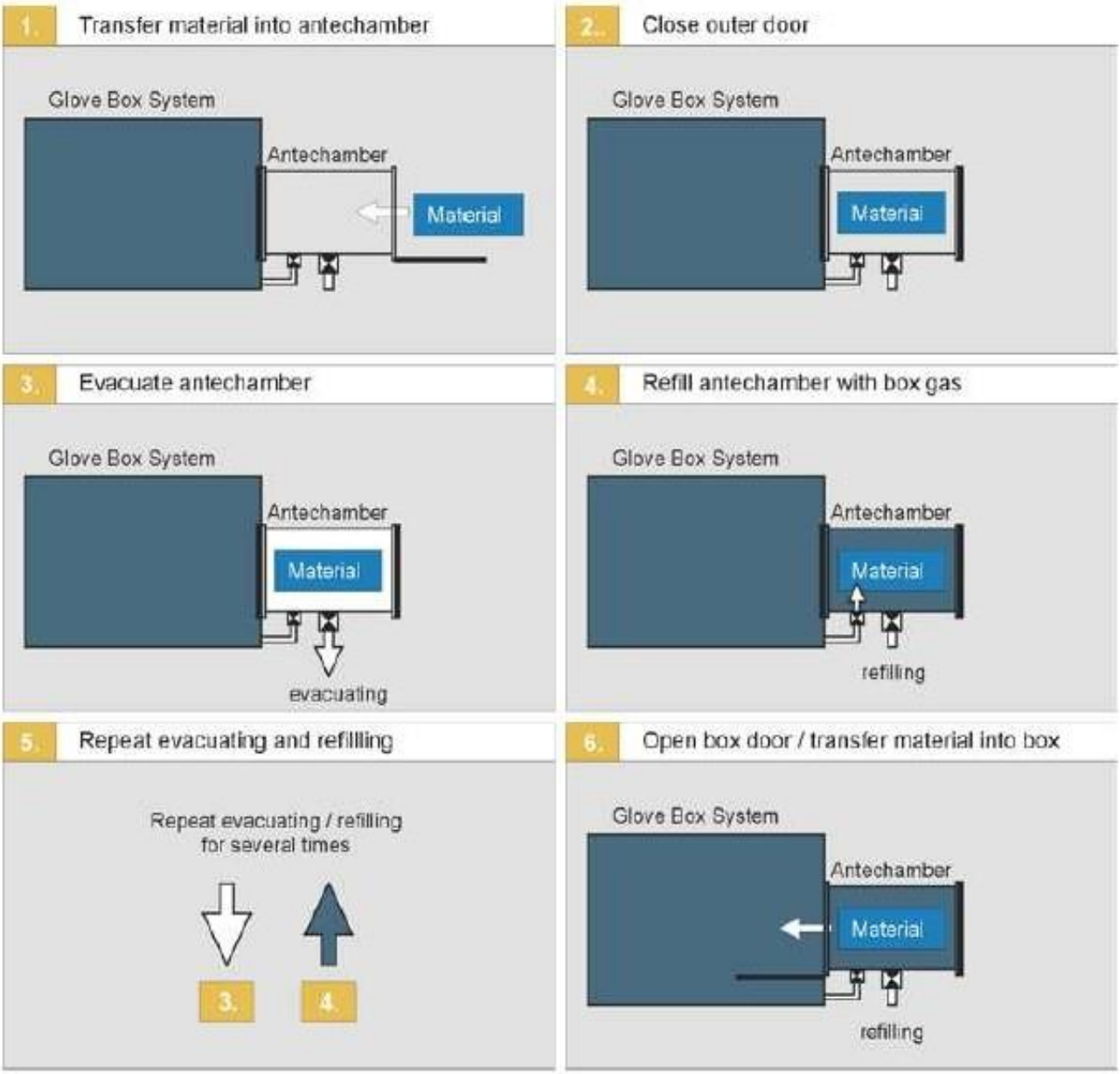
11. Note down the ppm level of O_2 & H_2O from the PLC in the log book before starting the process & after ending the process. Ideal levels should be O_2<<0.1ppm & H_2O<<0.1ppm. If the levels go beyond 50 ppm Quick Purge (described in Trouble shooting) the Glove box.
12. Make sure before putting your hand in the Glove box you have removed all accessories from your hands like watch, Rings, bangles etc. and you must wear cotton gloves so that your nails will not cut the gloves of GB.
13. Sample boxes and Petri dishes also should be taken out after experiment.
14. Minimum quantity of solvent should be used inside the box, Make sure that the quantity is entered in the log book

15. At present we are using ODCB, Chloroform and Xylene as solvents inside the glovebox. Any other solvent if need to be used inside the box, get the permission from FIC.

Spin coater and hot plate related guidelines:

1. Check the logbook and make an entry in the logbook before starting the process.
2. Put your sample box inside the antechamber and evacuate and refill it three times. After this take the sample inside the GB-1 (Make sure your sample box don't contain O_2 or H_2O. To ensure this make your sample box slightly open when you are putting it on the tray)

The procedure of taking sample inside through small antechamber is shown below:



3. The Solution vials containing solutions should be tightly closed with press cap and screw cap, in order to avoid spilling of the solvent while applying vacuum in the antechamber.
4. In the touch panel press **Box Light 1** it will make the tube light ON.
5. Then press the **Next** button in the touch panel, it will open another window.
6. Press the **Spincoater** button, it will change color from red to green. This will power ON the Spincoater control panel below the Glove Box.

7. Set the spincoater parameters like Speed, Ramp Rate and Duration before starting the experiment.
8. Open the *red valve* below the spincoater during the process of spinning and close it after use.
9. Press the low pressure footswitch while putting the hands inside.
10. Use gloves on top of glovebox gloves while working with solutions and resins (Epoxy).
11. Keep an aluminum foil in front of the spinner so that all the solutions are handled on the foil, and remove it after the experiment.
12. After putting the substrate on the center of the spinning table apply vacuum by pressing the **Vac** switch and check the pressure in the dial adjacent to the spinner table, the pressure should be near -1.0 mbar. If the pressure is not less than -0.5 mbar then the substrate is not attached correctly to the O-ring there or it is smaller in dimension.
13. It is always good to have a *Demo run* with a substrate before pouring the solution on the substrate to check the rotation speed and duration.
14. Now take the pipet and set the amount of solution to be sucked by the pipet by rotating the knob at its backside.
15. Attach a pipet tip on top of the pipet from the pipet tip box.
16. After opening the vial cap take the pipet and pressing the knob at the backside of the pipet enter the pipet tip in the vial and dip in the solution. Then slowly release the knob, it will suck the solution in the pipet tip.
17. Now slowly spread the solution on the substrate by pressing the pipet knob.

18. Then close the spincoater lid properly and start spinning.
19. After the spinning is done anneal the sample at a suitable temperature on the hotplate.

Note: The TARSONS Hotplate is capable of heating up-to 500ºC, but do not go above 200ºC inside GB-1 without written permission from FIC.

20. Temperature of the hotplate should be reduced after using it, and during the annealing, it shouldn't be left unattended.
21. Leave the hotplate at the minimum temp after the experiment. Once reached the minimum temp, switch off. Take care not to touch the hotplate when the temp is high. (It will melt the gloves)
22. Used micropipettes (wrapped in Al foil) are to be removed from the glove box after the experiment.
23. Solutions should be labeled if you are leaving them inside the box.
24. After experiment before taking sample out, evacuate and refill the chamber 3 times before opening the antechamber from inside and *leave the antechamber under vacuum at the end.*
25. Make entry in Hard copy and online log book.

Trouble Shooting:

Case: *O_2 level is high and O_2 purifier alarm is coming.*

What to do:

Quick Purge GB-1 through the following steps

1. Increase the box pressure to 6 mbar by pressing the high pressure paddle.
2. Switch OFF the **Analyzer** and **Circulation Purifier** from Touch Panel.
3. Then switch ON the **Quick Purge** by pressing the **Quick Purge** button, it will become green from red, you can hear the sound of purging.
4. Wait for 10 min. and then switch OFF the **Quick Purge** button by pressing it in the Touch Panel.
5. Then switch ON **Circulation Purifier** and wait for 1 min.
6. Then switch ON **Analyzer** and wait until the O2 level shows some stable value.
7. If the O2 level has not come down in an amount you want then do the Quick Purge again by following the same procedure as above.

Case: *Any unknown Alarm or Warning coming on the Touch Panel.*

What to do:

Check the 10th and 12th chapter of Glove Box manual and note down the error in the Log Book. Call System Owner or Lab In-charge to sort out the problem.

Case: *N_2 cylinder pressure goes down below 10Kg/cm^2*

What to do:

Change the N_2 cylinder through the following steps.

1. Increase the box pressure to 6 mbar by pressing the high pressure paddle.
2. Switch OFF the **Analyzer** and then **Circulation Purifier** from Touch Panel.
3. Close the regulator properly.
4. Then close the knob above the cylinder by rotating in the clockwise direction.
5. Then open the screw connecting the cylinder and the pipe by rotating it in counterclockwise direction.
6. Then replace the cylinder with a new filled one.
7. Connect the pipe to the cylinder and tighten the screw by rotating in clockwise direction.
8. Then open the knob above the cylinder by rotating in the anticlockwise direction.
9. Then open the regulator properly until the line pressure reaches around 6 mbar.
10. Switch ON the **Circulation Purifier** and then **Analyzer** from Touch Panel.
11. Wait until the O_2 level shows some stable value.

12. Also check the line pressure 3 min after the **Circulation Purifier** is ON. If it becomes less than 6 mbar then rotate the regulator in open direction until it become impossible to rotate it. Also open the knob of the cylinder more.