SOP

Glove Box-3 with Spin Coater and Hot Plate (Metal Halide Perovskite)

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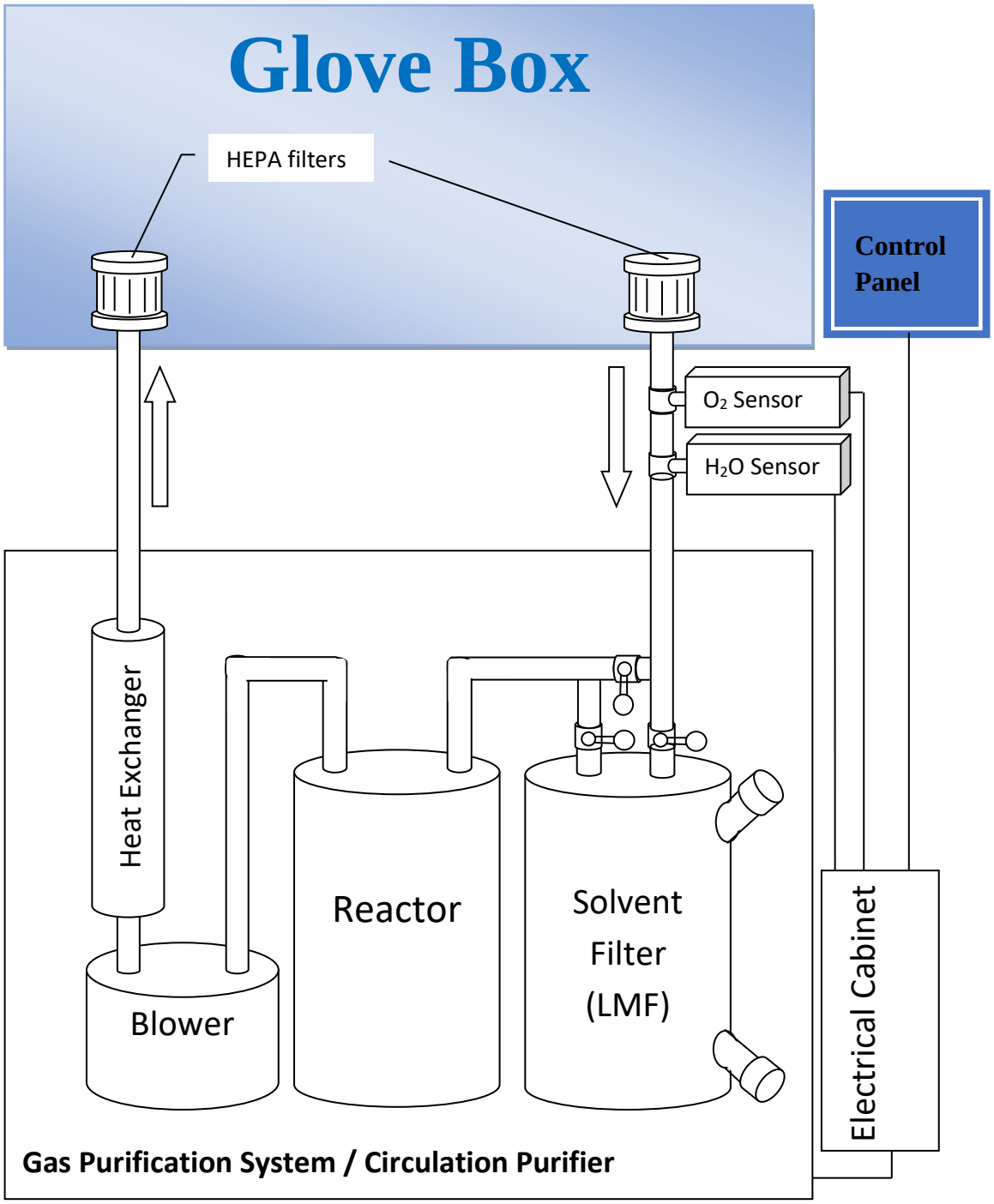
Introduction:

Generally, Glove Boxes are made for carrying out experiments in non-reacting ambient environment (in N2) . Experiments involving chemicals or materials that are reactive to the elements of the atmosphere can be done in Glove Box. **Glove Box-3 with Spincoater and Hotplate** (this is specially for Metal halid perovskite) has **N_2 ambient** inside the box for Spincoating of solution processed organic and/or Inorganic (or H_2O and O_2 sensitive) materials and to anneal them.
A schematic diagram of GB-3 can be depicted as shown below-



General instructions:

1. The first step is to check the availability and book a slot online through the CEN online portal.
2. Before entering the Organic Electronics Lab, wear the clean lab shoes and lab coat.
3. After entering the lab first activate your slot, put on the hair net, face mask and cotton gloves.
4. Make sure to check the previous entry in the GB-3 logbook before starting any process.
5. Check N_2 gas cylinder pressure. If it is less than 20 kg/cm^2 do not start any process inside GB & contact facility team (4402) to change the cylinder (contacts displayed in the lab).
6. Make sure the line pressure is greater or equal to 6 Kg/cm2.
7. Check whether the chiller and vacuum pump is ON (make sure by touching the surface of the chiller, and feeling the vibrations) and is at 18 ºC.
8. No aqueous solution is allowed inside the glove box.
9. 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-3 with a *high caution* and *covering the black gloves with* blue ones.

10. The box is controlled by PLC with Touch Panel and in Touch Panel, **Circulation Purifier**, **Analyzer** and **Vacuum Pump** should permanently be switched ON.
11. Samples Processed outside the GB should be taken inside through small antechamber with 3 times evacuation and refill cycle. Also, please take care that while taking the sample box or any empty vial kept the vial open to avoid unnecessary O2/moisture entering inside the GB-3.
12. Make sure all the things needed for the process are there in the GB-3. Such things are:
    i. Pipet and Pipet tips of the size you require
    ii. Tweezer
    iii. Blue Extra Large Gloves
    iv. Al foil

NOTE: Always try to arrange the things you want to enter in the GB-3 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-3.*

13. 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.

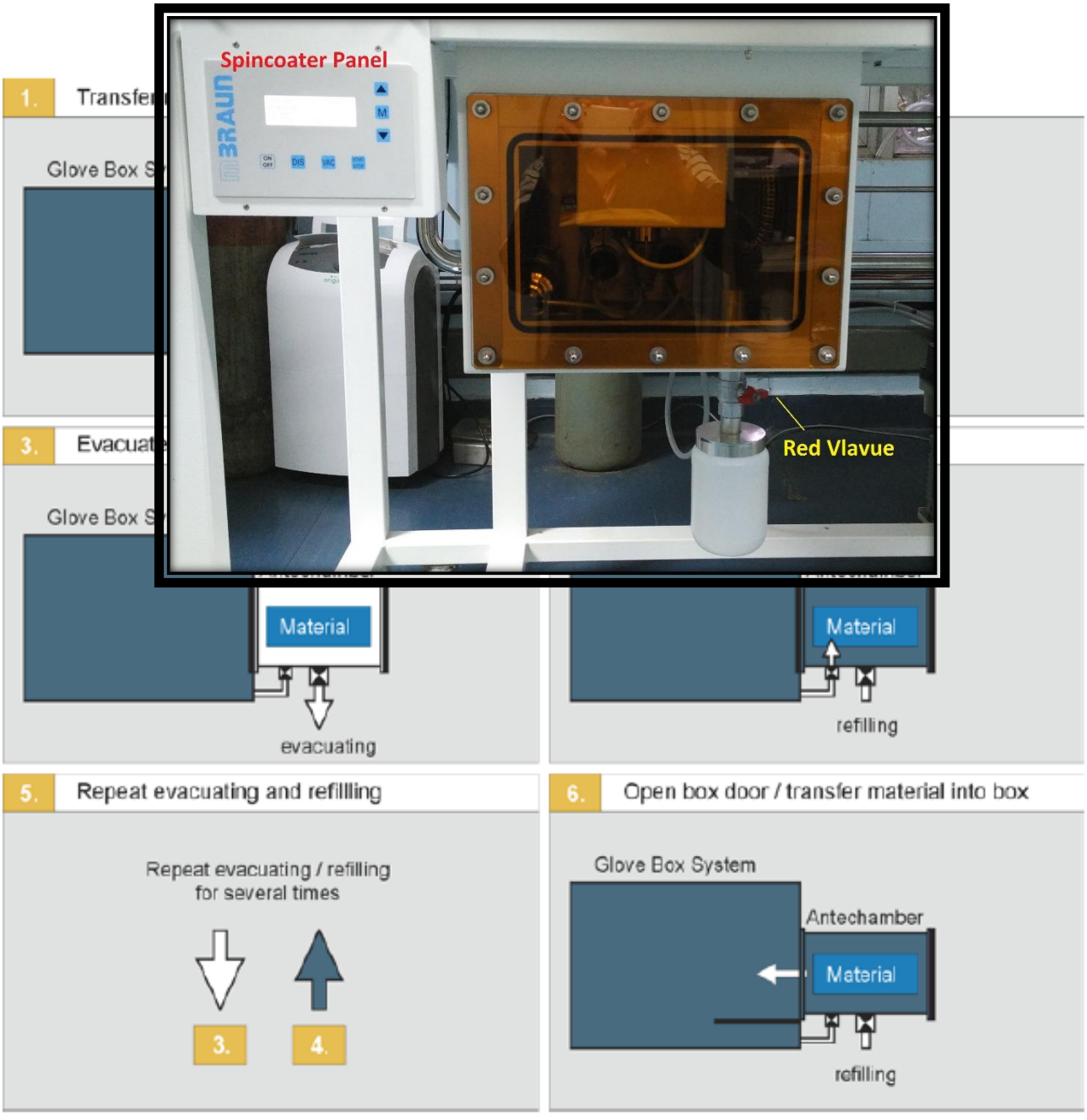
14. Make sure before putting your hand in the Glove box you have removed all accessories from your hands like watch, Rings, bangles etc. and you must wear cotton gloves (Full sleeves and it should not be torn) so that your nails will not cut the gloves of GB.
15. Sample boxes and Petri dishes also should be taken out after experiment.
16. Minimum quantity of solvent should be used inside the box, make sure that the quantity is entered in the log book
17. At present we are using ODCB, Chloroform, DMSO, DMF, CB, Hexane, Octane, and Xylene as solvents inside the glovebox. Any other solvent if need to be used inside the box, get the permission from FIC.
18. For taking any equipment or objects for specific purposes, that are not mentioned in the SOP, take written permission from the FIC and submit a copy of the same to any of the system owners, for records.

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-3 (Make sure your sample box doesn'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.
   If you want to take the gloves inside of GB then keep the gloves in small antechamber for 15 minutes in vacuum to remove the moisture. Don't take gloves and solution simultaneously because if you keep the gloves for 15 minutes in vacuum then during that time the solution might come out from the vials due to very low pressure.
4. In the touch panel press **Box Light,** it will make the tube light ON.
5. Power ON the Spincoater Panel by pressing ON in the panel.
6. Set the spincoater parameters like Speed, Ramp Rate and Duration before starting the



experiment.

7. Open the *red valve* below the spin coater during the process of spinning and close it after use.
8. Press the low pressure footswitch while putting the hands inside.
9. Use gloves on top of glovebox gloves while working with chemicals (dry, solvent and spin coating process).
10. 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.
11. 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.
12. 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.
13. Now take the pipet and set the amount of solution to be sucked by the pipet by rotating the knob at its backside.
14. Attach a pipet tip on top of the pipet from the pipet tip box.
15. 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.
16. Now slowly spread the solution on the substrate by pressing the pipet knob.
17. Then close the spin coater lid properly and start spinning.
18. After the spinning is done anneal the sample at required temperature on the hotplate.

Note: The TARSONS Hotplate is capable of heating up-to 500ºC, but do not go above 200ºC inside GB-3 without written permission from FIC.

19. Temperature of the hotplate should be reduced after using it, and during the annealing, it shouldn't be left unattended.
20. 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)
21. Used micropipettes (wrapped in Al foil) are to be removed from the glove box after the experiment. If solution spilt than clean before leaving the system
22. Solutions should be labeled if you are leaving them inside the box.
23. 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.***
24. Make entry in Hard copy and online log book.
25. We never do spin coating of PEDOT:PSS inside of GB's because PEDOT:PSS is a solution that is prepared with water.

Trouble Shooting:

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

What to do:

Quick Purge GB-3 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.

Case: *N_2 cylinder pressure goes down below 20Kg/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.

Important Rules:

1. Don't leave the hot plate ON for overnight or else be present for that period of time.
2. If O_2 level goes all high, do Quick Purge and inform the system owner.
3. Switch OFF all electrical instruments before leaving lab.
4. When there is a sudden power failure, then turn off the main switch (round black color).
5. Process to Shut Down the system:
    i. Increase box pressure
    ii. OFF analyzer
    iii. OFF circulation purifier
    iv. Close main switch
6. Process to start the system:
    i. On the main switch
    ii. Wait for few minute, then ON circulation purifier.
    iii. On analyzer.
7. Do not use hot plate temperature above 200^0C.
8. Do not keep hot plate ON after use.
9. Annealing process should be in slot bookingIR lamp process should be include in SLOT Booking
10. User want to use GB more than 15 min than user have to book slot
11. Big Anti-chamber shall be opened only during lab cleaning. So to take any big object inside wait for the lab cleaning day and plan accordingly.

If user perform some new experiment in GB then please take permission from FIC