

# **Standard Operating Procedure Glovebox and Thermal Evaporator**

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## Purpose of SOP

The purpose of this SOP is to describe the processes involved, and the care that should be taken, with both the spincoater and thermal evaporator in the POWER Lab glovebox. This will also describe troubleshooting measures to undertake when an error appears on the glovebox touchscreen Simatic panel.

## Personal Protective Equipment

EYE PROTECTION: Safety glasses or goggles

PROTECTIVE CLOTHING: White cotton gloves; Roll up sleeves of shirt; NO LABCOAT, RINGS, WATCHES, OR BRACELETS.

## Use of Liquid Nitrogen Tank to Provide Nitrogen Flow to the Glovebox

In order for the glovebox to function and maintain an air-free environment, a constant flow must be coming from the liquid nitrogen tank into the glovebox. In order to connect the tank to the glovebox, connect the pressure gauge coming from the box to the gas outlet of the tank. Make sure the gauge is set to 90 PSI in order to provide a flow capable of supporting the glovebox.

Liquid nitrogen tanks can be filled up outside the Physics Building at 9 am every weekday, in addition to 2 pm on Fridays. Ensure there is a full backup tank at all times in the lab. If you switch the line over to the backup tank, please let the liquid nitrogen superuser, Ryan Mulvenna, know so that the empty tank can be refilled.

Typically, it will take a full day for the liquid nitrogen tank to sufficiently pressurize in order to power the box.

## Bringing Materials into the Glovebox via Antechamber Doors

### **Side Antechamber Doors**

General:

- Before you bring anything into the glovebox, make sure that the antechamber has been refilled and the handle is in the neutral position. Furthermore, check to see if the antechamber door is fully closed inside the glovebox. Once you have placed your materials inside the chamber, close the door, and slowly pull vacuum. Leave the handle at the vacuum position for 3 minutes. After these 3 minutes, slowly refill the chamber. Repeat this process three times. On the last cycle, refill the antechamber, and place the handle in the neutral position. Carefully open the door and place your materials inside the box.

Closed Lids:

- If you are bringing in a material with a closed lid, make sure the lid is only loosely placed on top of your container. This will prevent the container from breaking.

Solvents:

- If you are bringing in a solvent or a sealed container of liquid, the process is different. Place the sealed container inside the antechamber. Then quickly evacuate the chamber until the pressure gauge reads -50 kPa (halfway). When you reach this point, quickly refill the chamber. Repeat this process 20 times. This is called a soft purge.

Other:

- Use the log book to note what you brought in and what the O<sub>2</sub> level was after finishing.

### **Large Antechamber Door**

If your material is too large to be placed in one of the side antechamber doors, you may use the main antechamber door in the middle of the glovebox. First, make sure the doors inside the glovebox to this chamber are closed. Then, using the Simatic touchscreen panel, press the button marked Antechamber. Refill the antechamber and wait for the pressure gauge to indicate it is at atmospheric pressure. Open the door and place your material inside. Close the door, and press the evacuate button to evacuate the chamber. Leave the chamber evacuating for 10 minutes. Proceed to refill the chamber once again. Repeat this process 3 times. Once you are done with three cycles, make sure the chamber has been refilled. Open the door on the side of the glovebox you want to bring your material into. Once you have brought your material in, close the door. Repeat the 3 cycles of 10 minute purging. This will ensure that anyone that is transferring materials between the two sides of the glovebox does not leak air in.

### **Glovebox - "Spincoater Side"**

The spincoater side of the glovebox is where any solvent related processes will occur. The hot plate on this side of the glovebox is intended only for the stirring of solutions.

#### Use of the hot plate

If you are using the hot plate, use one of the blue stirring holders to hold your vial on the plate. Make sure that the thermocouple sits firmly within the blue holder in order to maintain an accurate plate temperature. Please do not use heat on the hot plate if you are not using the blue holder/thermocouple.

If you need to heat a solution on the plate, leave a note on the glovebox window indicating your name, the temperature at which you are heating, and for how long you need the heat. If you need to heat up a solution, or cool down the plate, and see that someone is already using it at a different temperature, contact the person using the information left in the note. If there is something on the plate stirring at room temperature before you heat up the plate, contact the people who are stirring to make sure it is ok to take their sample off the plate.

If you are placing a sample on the hot plate, on the lid of your sample, write your initials and the temperature at which the sample should be stirred at. On the side of your vial, write what your sample is, and any other pertinent information to the sample.

Generally, the hot plate on the left side of the hood is kept at room temperature (unheated), while the plate on the right side is at 50°C.

#### Use of the mass balance

If you are using the mass balance, put on purple nitrile gloves (on top of the black gloves) in the box. Mass your samples using either a vial or the weigh paper. If you use a spatula, wipe the spatula with a kimwipe and chloroform. If it remains dirty, bring it outside the glovebox to clean.

#### Use of the spincoater

First, using the Simatic touch panel, press “Functions” and make sure the spincoater is on (green). Then, on the spincoater control panel (below the glovebox), hold the “on” button to turn on the system. Select the program you want to use. Put on purple nitrile gloves before handling your sample and solution. Place your sample on the spincoater chuck and press VAC. Add your solution to the sample and close the lid. Press Start to run your sample. When you are finished, place all materials you do not need in the trash container. Leave the gloves on the spincoater, unless they have become too soiled for reuse.

### **Glovebox-“Evaporator Side”**

#### Thermal Evaporator

Before using the thermal evaporator, you must be certified by the one of the superusers.

Before opening the evaporator door, using the Simatic panel, make sure the evaporator chamber is at atmospheric pressure. Use the layout panel to control the opening and closing of the shutter and thermal sources. Use the evaporator chamber panel to control the pressure.

Use the log book to record your evaporation each and every time. Include the material evaporated and the evaporation rates.

#### *Metal Evaporation*

Place the boat of your desired material into the evaporator in pocket 1. The boats for each metal are found in the labeled drawers to the right of the evaporator. If there are not any boats for the metal you wish to evaporate, additional boats can be found in the bottom drawers. Using the Deposition Controller panel, choose the process of the material you wish to evaporate. Place the substrate holder in the evaporator as taught by the superuser. When you are fully ready to evaporate, close the evaporator door, and press evacuate on the Simatic panel. Once the pressure is below  $4 \times 10^{-6}$  mbar, you can start your evaporation.

Press the start button on the Deposition Controller to begin the evaporation. Increase the power using the circular knob to 10%. Leave this at 10% for 5 minutes. After 5 minutes, increase the power to 15% for 3 minutes. After this, increase the power by 0.5% every minute until you reach your desired rate. If your material evaporates below 15%, start the 0.5% increase after 10%. Once you have achieved your desired rate, turn on the rotary holder on the Simatic panel to rotate the substrate holder. Open the substrate shutter and press zero on the Deposition Controller simultaneously to begin depositing on your substrate.

When you have completed your evaporation, power down in 5% intervals every 45 seconds until you are back down to 0%. On the Deposition Controller press Stop, Next, and then Reset. Refill the evaporator to atmospheric pressure. Once atmospheric pressure has been reached press the refill button again so it shifts from green to red. The evaporator may now be opened. Take down your samples and place the used boat into the corresponding drawer in the glovebox. USE ONLY ONE METAL PER ONE BOAT.

If a boat breaks, throw it in the trash container and use a new boat in its place.

### *Organic Evaporation*

Before you begin your evaporation, place the alumina crucible containing your material in one of the three organic sources. Make sure the crucible is approximately 1/3 full. Load the sample holder in a fashion similar to that in a metal evaporation. Close the evaporator door and evacuate the chamber to below  $4 \times 10^{-6}$  mbar.

On the Simatic panel, go the evaporator layout and press parameter. Press the source you placed your crucible in, and choose the material and temperature at which you want to evaporate. Once you have chosen this, go back to the layout screen. Press the source oven you have placed your crucible in to begin heating. Source shutters can be controlled in this layout menu.

When you have finished your evaporation, turn the source off. Let the temperature cool sufficiently before refilling the chamber. Take out your samples, wrap the crucible carefully in aluminum foil, and write the name of the material on the foil.

### Use of the Annealing Hot Plate

The hot plate on the evaporator side of the glovebox is intended solely for the purpose of annealing substrates. Do not use for anything else.

If you place samples on the plate, leave a note indicating the temperature at which you are annealing, and for how long you need to anneal for. This way, someone will be able to contact you if they need to use the plate.

### Trash Disposal

Each side of the glovebox has a container for trash. Place all your trash in this container. If the container is full, take the container out of the glovebox, and empty the container inside either the glass or lab trash depending on the material.

Once you have emptied the trash out, bring the container back into the box. If you are the person to fill up the trash, it is your responsibility to empty it.

### Sample Storage

If you are storing samples in the glovebox, place your samples in your own bin.

If you are using materials related to the evaporator, place each of the materials back in their respective bin when finished.

If you are using materials related to the spincoater or mass balance, place each of the materials back in their designated bins.

### Troubleshooting

#### *Purging the Glovebox*

If the O<sub>2</sub> level of the glovebox increases to greater than 0.1 ppm, you must take the following actions.

1. On the simatic touchscreen panel, press functions.
2. Turn off Circulation Purifier 1.
3. Turn on Quick Purge. Leave the purge on for 10-15 minutes.
4. Turn off Quick Purge.
5. Turn on Circulation Purifier 1.
6. Go to the Main Menu
7. Press LMF on the diagram.
8. Turn on circulation LMF Filter 1.
9. Check to see the O<sub>2</sub> level is below 0.1 ppm.

#### *Regenerating the Catalyst*

Every 6 months, the catalyst must be regenerated. First, hook up the H<sub>2</sub> gas gauge to an H<sub>2</sub> tank. Turn off Circulation Purifier 1, and turn on Regeneration Purifier 1. The regen will begin, and the entire process will take 17 hours.

Once it is completed, unhook the H<sub>2</sub> tank and purge the glovebox as described above.