CORAL Name: GB_Spincoater

Location: Lab 2U, 12-3111
Category: Spin Coating
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Introduction

The GB_Spincoater is an inert nitrogen glovebox that provides ambient conditions with less than 1 ppm levels of water (moisture) and 1 ppm oxygen (O_2). The glovebox is intended as an environment to enable the processing of oxygen and moisture-sensitive samples.The glovebox contains equipment to enable chemical processing of moisture or air-sensitive samples including:

- a spincoater,
- a microbalance,
- a vortexer for accurate mixing of solutions,
- a hot plate for baking samples after spin coating,
- various supplies.

Only anhydrous materials are allowed in the glovebox; this includes both solid materials and liquids. Porous solids like paper, Fabwipes, foam and fabric should not be brought into the Glove Box. Samples are introduced into the glovebox through an antechamber which is cycle purged multiple times to remove any residual moisture and to maintain a pristine processing environment.

Safety

The glovebox is not intended as personal protective equipment, its primary intent is to protect the materials and samples inside from exposure to oxygen or moisture. The doors to the antechambers may be pinch points, so use caution when opening and closing. Handling of materials and equipment through the gloves may be awkward, so work slowly and carefully to avoid knocking over or damaging equipment and samples. Forceps are available inside the glovebox to help grasp out-of-reach items.

The glovebox gloves provide an interface to handling items inside the environment, they are not intended as personal protective equipment (PPE).

- For all chemical processing, standard PPE (nitrile gloves) must be worn over the glovebox gloves and safety glasses must be worn at all time. Some chemicals may react with the butyl glovebox gloves

causing them to dissolve or soften, the disposable nitrile PPE protects them, extends their lifetime and prevents the spreading of contamination to other samples.
- Careful handling and proper disposal of sharps, including samples, is required. If the gloves are torn or punctured, seal with electrical tape and report in CORAL.
- General mechanical hazards such as squeezing, stretching, moving parts, sharp corners and edges exist when using this system. Be aware of those hazards and work carefully.

All personal chemicals must be reviewed and approved by MIT.nano Safety Staff. Submit a Personal Chemical Request on the linked form and staff will follow up. All chemicals and samples must be clearly labeled with a name and date, unlabeled items will be disposed of.

The chemicals used in this glovebox may be toxic or flammable. Follow all Wet Chemical Processing Guidelines and be aware of the materials that you are handling and how to treat an exposure. Safety Data Sheets (SDS) for all chemicals in the glovebox are available, observe relevant precautions when working with those chemicals. Spills may cause fire or explosion hazards during cleaning or subsequent use of the antechambers. Dispose of any waste at the end of each session, do not allow waste to accumulate. Be sure to add your waste chemical name to the collection tag as it is added to the container.

The glovebox has additional electrical and thermal hazards from the equipment inside.
- Do not attempt to modify any electrical wiring in the system or to defeat any safety interlocks.
- Do not unplug any electrical cords inside the glovebox.
- When using the hotplates, use caution to avoid burns or melting the gloves.
- Hotplates are for baking of samples only. DO NOT HEAT CHEMICALS directly on the hotplates.

When loading and unloading samples, a vacuum is pulled on the antechamber. Make sure any containers or vessels used for transfer are vacuum compatible to avoid breaking glass during this cycle purge sequence.

User Qualifications and Responsibilities

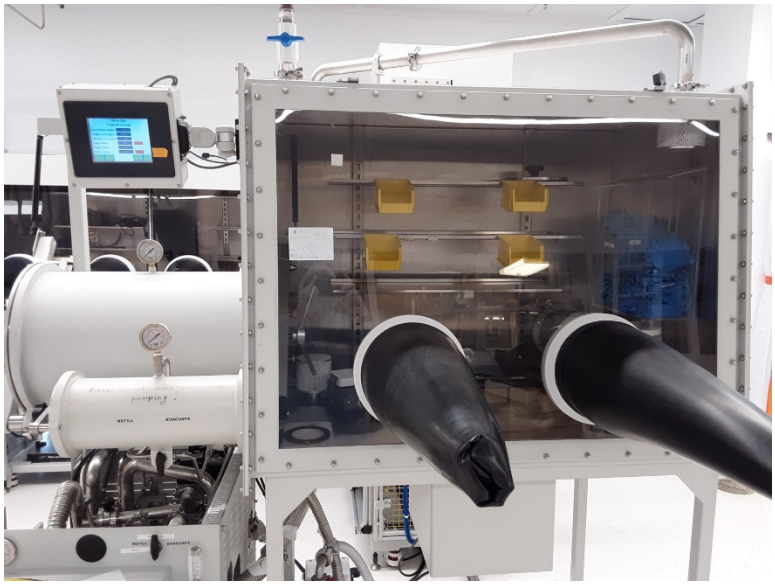
To maintain the low oxygen and low moisture environment, it is the Users' responsibility to observe the following rules and best practices. Failure to do so may result in disqualification and retraining will be required.
- All users must have an approved PTC process and all Personal Chemicals must have approval from MIT.nano Safety Staff.
- Users must receive training and qualification on glovebox use from MIT.nano Staff. Users must be engaged on the glovebox for the duration of their work.
- Abnormal conditions must be reported immediately in CORAL. This includes high O2 (greater than 1ppm) or moisture levels (greater than 10ppm), issues with the antechamber cycle purge process and issues with the gloves.
- Users must be familiar with the above Safety requirements and must follow all safe working practices in the lab.
- Users must pass a quiz on glovebox safety and best practices.
- At the end of each run both antechambers must be left under vacuum.
- Users must record the O2 and moisture levels at the start and end of each run in the log book (see Appendix).
- When processing with solvents, the circulation must be turned off. Failure to do so will poison the catalyst that purifies the environment and will result in extended downtime for system maintenance.

- At the completion of solvent work, once the chemical is capped again, the glovebox must be purged for >/= 30 minutes. The user should remain engaged in CORAL throughout the purge. This will help the user to remember to shut the purge gas off.
- The circulation (Blower) must be turned back on after purging is complete.
- The disengage the tool in CORAL.

Materials: Only **anhydrous** materials are allowed inside the glovebox. This rule applies to solids and solvents.
Porous materials such as fab wipes or cardboard (for boxed gloves, foil rolls) are not allowed as they can introduce moisture to the environment. If they are required, then staff must assist in order to prepare and introduce those materials properly.

Storage: Approved materials may be stored inside the glovebox. All samples and chemicals must be labeled with the username, the date, and the chemical name. Abreviations are acceptable if the chemical is added to "key" which provides full details and chemical names and is posted on the side of the glovebox. Materials dissolved in non-hydrocarbon solvents must be stored in **Teflon-capped vials with electrical tape wrapped around the rim.** Materials stored in any other way (unwrapped vials, centrifuge tubes, syringes, etc.) will be removed from the glovebox. When a user has finished with a chemical it should be removed from the glovebox and properly disposed of (MIT.nano Staff can assist).

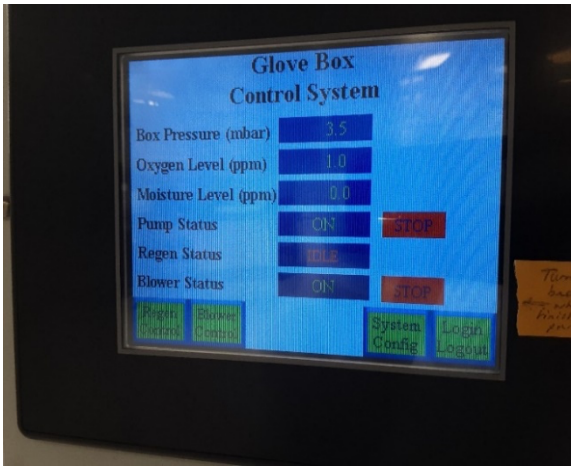
Operating Procedures

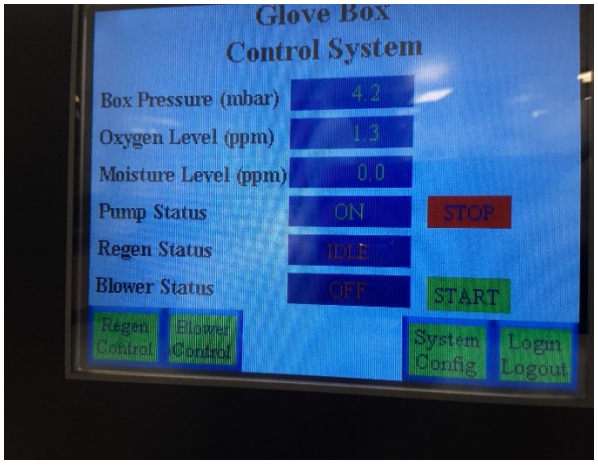


The Spincoater Glovebox is shown. The Control Interface is at top left. Antechambers are shown in the middle left. The purifier cabinet is at bottom left.

1) Check reservations in CORAL to ensure that you have reserved the correct machine at the correct time. Engage the tool in CORAL.

2) Open the **Small Antechamber** and load your samples, then close the door. Initiate the cycle purging sequence to minimize moisture coming into the glovebox: run **3 cycles x 5 minute pump-down** (15 minutes total).
   a) Use the manual “Refill/Evacuate” valve on the front of the antechamber to move back and forth between pumping and backfilling with nitrogen.
   b) Open Refill valve to fill the antechamber with nitrogen until it reaches atmospheric pressure
   c) Open Evacuate valve to pump down the antechamber for 5 minutes (use a timer)
   d) Repeat the above steps three times
   e) When not in use, the antechamber should be pumped down and isolated.
   f) Make sure all containers are vacuum-compatible: don’t introduce anything with trapped air into the glovebox (capped vials, syringes/needles in their wrappers, transfer tubes capped in air, etc.) Thin-walled glass or plastic containers can potentially explode when placed into a vacuum.
   g) For larger samples the **Large Antechamber** is available**:** run **3 cycles x 25 minutes** cycle purge (75 minutes total).
   h) For porous materials, pump down for at least 12 hours before cycle purging and bringing the material into the glovebox.
3) **Blower (Purifier):** The circulation blower must be turned off during the use of any solvents; failure to do this will poison the purifier catalyst and result in extended downtime for maintenance. To operate the circulator:
   a. From the “Main Screen” find **Blower Status** (indicated by ON/OFF in the blue box).
   b. Press the red box labeled **STOP** to turn if off.





4) **Purging:** Following any solvent work in which the circulation blower has been turned off, the glovebox must be purged for 30 minutes, after which time the circulation blower must be turned back on.
   a. Clean the glovebox work surface and remove all waste, including hazardous waste.
   b. Open the red Purge valve on the top left of the glovebox to start the purge process, start a timer for 30 minutes.
   c. Close the red Purge valve to stop the purge when time has elapsed. NOTE: purging uses a lot of nitrogen, make sure to stop the process before disengaging from CORAL and walking away.
   d. On the “Main Screen” next to **Blower Status** press the green box labeled **START** to turn the blower back on.
5) At the end of all processing transfer samples back into the antechamber and make sure the internal door is closed. Remove samples and waste from the antechamber.

6) Ensure all remaining equipment is turned off and that the glovebox is clean and that all samples, waste, and materials have been removed.
7) Pump down the antechamber: open the Evacuate valve until the gauge reaches -27 inHg, then close the valve.
8) Disengage the tool in CORAL and accurately report details in the required fields.

Appendices

Spin Coater Operation

The Spincoater and its control panel are located in the well under the deck of the Spincoater glovebox. The coater can store up to 30 recipes with 20 steps in each recipe.

1. Open the vacuum valve at the backside of the spincoater well. The handle is black plastic.
2. Remove the deck sections and store them along the side of the glovebox while using the coater.
3. Remove the controller from the well and place it in a visible location.
4. Line the spinner bowl with aluminum foil. This will collect excess material and be disposed of as waste at the end of the process.
5. Place your sample on the chuck. The vacuum will engage once the recipe starts.
6. Use the UP and DOWN arrows to step through the various saved recipes.
7. Once your desired recipe is displayed, press the **MODE** button to enter the Program/Edit mode.
8. A cursor > will point to the recipe number.
9. Press **ENTER** to move through the different parameters within a step.
10. Spinner parameters are shown in the following table. Press the UP and DOWN arrows to adjust values, then press ENTER to save the new values before moving on to the next parameter.

STEP	RANGE (Up to 20)	DESCRIPTION
RAMP	0 to 25.5 seconds	Time to accelerate from initial speed to new speed.
SPEED	0 to 9999 RPM	The spin speed of current step.
DWELL	0 to 999 seconds	Time for the current step at above speed.
DISP	NONE	Dispense option, not available on this spinner.
TIME	0 to 10 seconds	Dispense time, not available.

11. Press the MODE button again and the handset will display RUN.
12. Press START to run a test spin to make sure the sample is centered, adjust as needed.
13. Dispense the material to be spin-casted, then close the lid.
14. Press START to run the process.
15. During operation, the display shows step, speed and time remaining.
16. At the end of the process, the screen will display REMOVE COATED PARTS. Now your sample can be removed.
    a. Press STOP at any time if you wish to stop the recipe.
    b. Press the CLR ERROR button if an error occurs. Press START to restart the recipe from the beginning.
17. Carefully collect the aluminum foil liner, folding it in on itself to avoid spreading chemical contamination. Move waste into the antechamber for disposal.
18. Replace the spinner lid, put the spinner controller back into the well and replace the deck section.
19. Turn off the vacuum valve at the back of the spinner well.

Tips, Tricks and Best Practices

- In normal operation when the glovebox is under positive pressure, the gloves will be extended outside of the box.
- Drooping gloves during purging steps is normal. If this is observed at other times, report in CORAL.
- If a necessary solvent cannot be obtained in anhydrous form, then details should be discussed with staff. Total volume must be minimized to avoid contaminating the chamber with moisture. The container must be tightly sealed and taped when not in use.
- Cycle Purging of the antechamber must be done every time it opens to atmosphere.
- Move your arms into the glovebox slowly. Avoid abruptly extending the gloves into the glovebox as this will result in a pressure spike/pulse that can result in contamination.
- Avoid using sharp tools and samples as much as possible.
- Safety covers, panels, panes, windows or doors may not be removed at anytime by users.
- It may be easier to see the spin coater display with the glovebox lights off.
- If you attempt to use the spin coater and see a VACUUM FAULT, it is because the vacuum valve is not open.
- Record data on the log sheet (see Appendix) and in CORAL so that system health can be accurately tracked.
- To bring new, user-specific items (such as LCD screens or adhesives) into the glovebox, users must consult staff for best practices.

Glovebox Usage Logsheet

Date	User	Time In	H2O/O2	Purge Start	Purge End	H2O/O2	Solvents	Notes