Glovebox Safety Considerations

User and System Safety

The MBraun glovebox presents relatively few safety hazards to the operator due to its built in safety controls (e.g., if the glovebox senses an overpressure >15 mbar, it automatically shuts off gas flow to the box to prevent blowing out the gloves or shattering the window). Issues such as excessive box pressure or elevated water (H_2O) or oxygen (O_2) levels will generate "Alarms" indicated by a flashing red "Alarms" box at the bottom left of the glovebox touchscreen controller. Pushing the "Alarms" box on the touchscreen will take you to the Warning Screen where you can push the "ACK" button at top right to acknowledge any active alarms and turn them off. Note, however, that *some alarms also require further action on the part of the user* (e.g., if gas recirculation or the H_2O/O_2 analyzers have been shut off) as described in greater detail in the Glovebox Damage Prevention section of this document.

Because our system is used for sodium and lithium ion battery electrode studies, *the inert atmosphere must be composed of argon (Ar) rather than nitrogen (N_2).* The biggest safety concerns surround the high pressures associated with the compressed gas cylinders that supply the inert working gas (Ar) atmosphere and flammable regeneration gas (3-10% hydrogen in Ar). It is important to ensure that the glovebox exhausts are properly connected to the building ventilation system in order to vent any potentially harmful vapors removed from inside the glovebox. In addition, the usual electronic instrumentation safety concerns apply – electrical shock. To minimize these risks (as well as the potential for damage to the system), operators should not open the gas purifier chassis, AFM controllers, or computer without permission from the lab manager.

Glovebox Settings

Typical glovebox settings and standard procedures:

- Allowable box pressure range is user adjustable (via touchscreen) within ±15 mbar range. Defaults values are:
    - Minimum = +1 mbar
    - Maximum = + 6 mbar
    - Hysteresis = 2 mbar
- Gas regulator outlet pressures
    - Inert atmosphere = 80 psi
    - Regeneration gas = 5 psi
- Gas cylinder status
    - Always ensure signage on cylinders (Full/In Use/Empty) is correct.
        - A new (full) gas cylinder should have a pressure of >2600 psi, and will need to be marked as "Empty" and replaced when it drops below 100 psi.
    - Record cylinder pressure in logbook daily ***and*** at each use, also noting water (H_2O) and oxygen (O_2) levels in the box (should be <0.1 ppm). This is important for tracking glovebox performance over time, detecting issues (e.g., leaking/damaged gloves, reduced gas purifier performance, lack of gas recirculation, etc.) in a timely manner when they occur (i.e., *before* they cause damage to the system or samples), and identifying the source(s) of increased water or oxygen levels.

        - Swap out the inert gas supply tank when it drops to 100 psi (just enough to maintain 80 psi outlet pressure), but notify the SSL manager if it's at 500 psi or less so that he can plan accordingly.
        - There should always be a spare full inert gas tank. If you empty the tank currently in use and swap it out for the spare tank, let the SSL Manager know so that he can order a replacement spare.
- Gas analyzers
    - The H_2O and O_2 analyzers should be turned *on at all times except during regeneration or analyzer sensor cleaning*.
        - If you notice the analyzers are turned off (no H_2O and O_2 levels indicated at the top of the home screen or the "Analyzer" box is white rather than green), turn them back on immediately.
        - H_2O and O_2 analyzers can be turned on by either of two ways:
            - Push the "Functions" button on the left of the home screen, then push the "Analyzer" button in the middle of the right column on the resultant screen to turn it green.
            - Push the rectangular VPG/RKM/GB1 indicator box on the bottom of the home screen, then push the "Analyzer" button toward the bottom of the column on the right of the resultant screen to turn it green.
    - H_2O and O_2 levels should typically stay at <0.1 ppm except for occasional small temporary spikes upon running an antechamber cycle, pulling gas from the cylinder to automatically refill the box (due to diffusion of H_2O and O_2 through the Tygon tubing), and opening or introducing samples into the glovebox.
- Gas recirculation and purification
    - To protect the gas purifier, *always purge (rather than recirculate and use gas purifier) if O_2 is >100 ppm!*
    - **Important**: if the box senses an overpressure >15 mbar, it automatically shuts off both the H_2O and O_2 analyzers as well as all gas flow to the box (both from the gas cylinders and recirculation through the gas purifier unit). In addition to acknowledging the resultant alarms, you will therefore need to ***reactivate the analyzers*** (see section above) and ***turn the circulation back on***.
        - At the bottom of the home screen, the rectangular VPG/RKM/GB1 indicator box will show both the RKM (gas purification unit) and GB1 (glovebox gas inlet valve solenoid) indicators as white (off/inactive) rather than green (on/active).
        - There are two ways to turn back on gas recirculation/purification:
            - Push the "Functions" button on the left of the home screen, then push the "Circulation Reactor 1" button at the top of the left column on the resultant screen to turn it green. Make sure the "Analyzer" button is also green.
            - Push the rectangular VPG/RKM/GB1 indicator box on the bottom of the home screen, then push the "Circulation" button at the top of the column on the right of the resultant screen to turn it green. Make sure the "Analyzer" button is also green.

    - Increasing contaminant (O_2 and/or H_2O) levels with time is indicative of the need to regenerate the gas purifier. If this is the case, stop using the glovebox and notify the SSL Manager.
- Box history (pressure as well as O_2 and H_2O levels for the last 24 hours) is accessible via the "Box Trend Curves" option on the touchscreen.
    - A sawtooth pressure pattern is indicative of a leak and should be reported to the SSL Manager.
        - This could be caused by an improperly sealed antechamber door, leak in the gloves, or the AFM stage vacuum being left on inadvertently.
    - Large spikes in O_2 or H_2O, especially upon introduction of something via the antechamber, can be indicative of an insufficient pump-purge cycle.
- Exhausts and ventilation
    - Ensure all exhausts (vacuum pumps, gas purifier regeneration, and box purge) are all properly connected to the building exhaust ventilation system (PVC pipe in SE corner of SSL next to gas cylinders). This is of particular importance when regenerating the gas purifier or purging the glovebox.

Glovebox Damage Prevention

Portions of the glovebox can be easily damaged by inattentive operation, resulting in significant repair costs. Additionally, the glovebox atmosphere can be compromised, leading to destruction of samples, poisoning of the gas purification system, and/or safety hazards due to release of toxic vapors or ignition of pyrophorics. As a result, users should familiarize themselves with the ways in which the system may be damaged *before* operating the glovebox or associated AFM. To become certified on the system, users must be familiar with the good practices described in the sections that follow.

Antechambers

The MBraun glovebox has two antechambers, the standard (large, ~15" diameter) antechamber, and a smaller (~6" diameter) mini antechamber. The former is evacuated by a standard rotary vane oil-based pump, while the latter employs a rotary vane roughing pump to back a turbomolecular pump. For most samples, the mini antechamber is sufficiently large and pulls much less Ar from the box to refill. It also reaches significantly lower pressures due to the turbo pump, can evacuate much more quickly, has a highly accurate vacuum (pressure) gauge, and is easier to open and close. The mini antechamber is therefore preferable. Important considerations:

- *Never attempt to evacuate an antechamber with a door open!*
- *Never open the door to an antechamber when it's actively being refilled (i.e., Refill is green), even if it's at atmospheric pressure, as this can lead to introduction of oxygen and water into the glovebox!*
- *Likewise, never open both doors (interior and exterior) on a given antechamber at once!*
- *Don't overtighten the clamp on the large antechamber doors!*
    - Over time, this can pull the antechamber door away from the glovebox wall, thereby compromising the seal.
    - Simply spin the door clamp with a single finger until it's snug.
    - Never tighten the door clamp while the antechamber is under vacuum – you won't be able to open it once the antechamber is pressurized.

- When closing the mini antechamber door, check to ensure the O-ring gasket causes the upper lip of the door to point slightly away from the antechamber itself.
    - If not, the O-ring gasket is overly compressed and needs to be rotated or replaced.
- The turbo pump attached to the mini antechamber requires both the roughing pump and the water chiller in order to operate.
- Large items and/or those likely to off-gas for a significant period of time (e.g., paper products, gloves, foam, etc.) should typically be placed in the antechamber and stored under vacuum overnight, followed by a standard pump-purge cycle.
- Containers should be left ajar/cracked open for air flow during pump-purge cycles.
- Do not use/put your hands in the glovebox gloves during the mini antechamber's pump-purge cycle.

Gloves

- Inspect the gloves for cuts, nicks, or scratches at each use and notify SSL manager if they need to be replaced ($119/pair).
    - Leaking gloves often result in a sawtooth pressure pattern in the "Box Trend Curves".
- To prevent damage, *remove any watches or sharp jewelry (e.g., diamond rings) prior to inserting your hands into the gloves*.
- Wear cotton liner gloves inside the glovebox gloves to keep them clean for other users.
- Put clean disposable nitrile gloves on over the glovebox gloves (i.e., inside the glovebox) to improve manual dexterity and protect the glovebox gloves.

Glovebox AFM

- *Always remember to turn off the AFM stage vacuum (switch on the front panel of the AFM) to prevent gradually draining the glovebox atmosphere (and working gas supply tank).*
- Don't leave items on the granite AFM stage where the sample chuck can run into them and potentially get stuck or damage the precision motor.
- Both the AFM electronics (including the piezos) and many of its accessories (e.g., SECM probes, which cost ~$500 apiece) are *highly susceptible to damage from electrostatic discharge (ESD), which is exacerbated by the extremely dry atmosphere inside the glovebox*. To **prevent ESD**, always:
    - Stand on the anti-ESD anti-fatigue mat on the floor in front of the glovebox (and make sure its grounding wire is connected to the grounding bar on the wall of the SSL).
    - Attach the anti-ESD heel strap kept on top of the gas purifier box next to the glovebox logbook.
    - Utilize the workstation ionizer inside the glovebox, ensuring it's within 12" of the items you're handling.
- Run the glovebox blower (recirculation) fan at 65% for optimal flow (minimal turbulence) and acoustical noise.
- The AFM heads (especially the FastScan head) are extremely heavy and difficult to move or swap inside the glovebox. Additionally, *the AFM service contract does not cover damage due to dropping the heads*.
    - *If you need to swap AFM heads, consult with the SSL Manager.*