

Glovebox Maintenance SOP

MBraun MB20 Gas Purifier Unit and LabMaster Pro 3 Glove Glovebox with TP700 Touch Panel

General Notes

- All user manual references in this SOP are to PDF Manual Version 4.1.
- Always run the glovebox blower fan at 65% for low airflow/currents and minimal AFM noise.
- A good operating pressure range is 1-6 mbar with 2 mbar hysteresis.
 - Since the outlet pressure of the Ar feed tank is set at 80 psi, swap out the tank when the cylinder pressure drops to 100 psi.
- Antechambers and pumps:
 - For the large antechamber, 3x 5 minute pump/refill cycles is typically sufficient unless pumping overnight on something that offgasses significantly.
 - Operation of the mini antechamber's turbomolecular pump requires cooling water from the chiller located in the mechanical chase.
 - If you see a large (>100 ppm) spike in O₂ or H₂O, turn off circulation and begin purging to avoid saturating the gas purifier.
- The Lexan glovebox window can be cleaned with Windex.

Absolute No-Nos

- Overtightening the clamp on the large antechamber doors (should just be fingertight at atmospheric pressure).
 - Tightening the clamp on the large antechamber door while under vacuum.
- Opening both doors on an antechamber at the same time.
- Evacuating an antechamber with a door open.
- Rings, watches, etc. inside the glovebox gloves.
- Leaving things on the AFM's granite stage (where the AFM stage can hit them and/or get stuck).
- Forgetting to turn off the stage vacuum (this will drain the Ar tank).

Recommended Routine Preventive Maintenance

- Clean the H₂O sensor every 2,000 hours
 - Alarm on touchscreen will indicate when hours are reached
 - Need DI water and phosphoric acid (3 drops of 80%)
 - Check centering ring for dry rot (or swap out) each time
- Recalibrate the O₂ sensor on an annual basis
 - \$650 with 6 week downtime
 - \$1050 if it requires repair

- \$4200 for a spare sensor
- Gloves are \$119/pair
 - Always sold as a pair - either a left and right or 2 ambidextrous gloves
 - Replace gloves and glove O-rings (2 per glove @ \$10/O-ring) annually
- Replace door O-rings annually
 - \$15 apiece (x2) for large antechamber
 - \$9 apiece (x2) for mini antechamber
- Replace HEPA filter annually
 - \$80
- Replace carbon in trap every other month
 - \$37.50 for 1.25 kg (one refill)
 - Replace paper filter (\$50) every other carbon change (i.e., every 4 months or 3x/yr)
- Replace vacuum pump oil whenever a regeneration is run (every 4-6 months, or when O₂/H₂O levels start to increase 4-5 ppm/hr, or ~0.6 ppm/5 minutes)
 - Oil is \$20/bottle

Historical Notes

- System installed April 2, 2018 (2018-04-02)
- Right hand glove (closest to antechambers) replaced May 24, 2021 (2021-05-24)
- MBraun preventive maintenance (PM) visit on July 9, 2021 (2021-07-09)
 - Flipped and lubricated antechamber O-rings
 - Replaced HEPA filter (inside top right of glovebox)

H₂O Sensor Cleaning

- Alarm on touchscreen will indicate when 2,000 hours are reached.
 - Probably only needs to be performed every 4,000 hours (i.e., acknowledge alarm and clean sensor every other time, or approximately 2x/year).
- Ensure you have DI water and concentrated phosphoric acid (3 drops of 85% H₃PO₄) on hand, along with KimWipes or lens tissue.
- Using the touchscreen (under Menu | Box Pressure), increase the Upper Working Limit to its maximum value of 14.5 mbar and pressurize the glovebox using the foot pedal.
- Turn off Circulation and Analyzers using the glovebox touchscreen.
- Unplug, then quickly remove the sensor and cap the flange with a KF-25 blank.
- Check the centering ring for dry rot.
- Unscrew the protective screen around the sensor.
- Rinse the sensor with DI water and thoroughly pat dry, taking care not to leave any residue on the sensor.
- Add a minimal amount of phosphoric acid to fully wet the surface of the sensor (1-2 drops)
- Reattach the protective screen and put the sensor back on the glovebox.
- Turn on Circulation and Analyzers using the touchscreen.
 - Decrease the Upper Working Limit back to its normal value of 6 mbar.
 - Monitor the water and oxygen levels, which should start to come back down quickly (2-3 minutes for oxygen; 15-30 minutes for water).
 - Note that it may take several hours for water levels to return to <0.1 ppm if too much water was left on the sensor and/or too much phosphoric acid was used to wet it. Gas purifier status can also impact this.

Gas Purifier Regeneration

- Touchscreen Menu Functions
 - From the Main/Home screen, select the "VPG/RKM/GB1" rectangle at the bottom (p. 154, Sxn 7.3.1)
 - Select Parameter from the lefthand menu (p. 155 and 158, Sxn 7.3.1 and 7.3.1.1) and check that:
 - Blower speed is set to 65% (best AFM performance).
 - Regeneration gas H₂ content should be selected as $\geq 5\%$ (normal), as the nominal H₂ content from Norco is 5%.
 - Accordingly, the purge time should automatically set itself to 150 minutes (2.5 hours).
 - If you use more dilute regeneration gas (<5% H₂ content), the purge time increases to 300 minutes (i.e., 5 hours).
 - The "Automatic Circulation" parameter should be set to "Yes" so that recirculation automatically restarts as soon as the regeneration cycle is completed.
 - Total time for regeneration is 16 hours (with $\geq 5\%$ H₂ content), so if you start at 5 PM, it will be done by 9 AM the following morning.
 - Select Status from the lefthand menu (p. 155 and 159, Sxn 7.3.1 and 7.3.1.1); from this screen, you can see:
 - Remaining regeneration time in minutes (if there's currently an active regeneration cycle)
 - Time since last regeneration (in hours)
 - Total time the purifier's been running since the system was installed (in hours)
 - To run a regeneration cycle (p. 166-167, Sxn 7.3.2.4):
 - Toggle off the green "Circulation" and "Analyzer" buttons so that they're white (p. 155).
 - Toggle on the white "Regeneration" button so that it's green.
 - You'll be asked to confirm that you want to run a regeneration cycle
 - Select "Yes"
 - You'll then be asked to confirm the regeneration gas flow.
 - Go into the mechanical chase and open the valve on the top of the regeneration gas cylinder (if not already open) as well as the outlet valve on the regulator.
 - Check that the outlet pressure is ~6 psi with both valves open; if not, adjust the outlet pressure to achieve the desired value.
 - Check the flow regulator on the bottom right front of the gas purification unit; it should register a flow rate of 15-20 L/min, lining up with the double arrows on the flow regulator.
 - If everything looks good, click the red confirmation button, and the regeneration cycle should automatically start.
 - After a regeneration cycle, the recirculation should automatically restart, but you may have to turn the analyzers back on.

Vacuum Pump Oil Change

Procedure

- Oil change procedure
 - Ensure antechambers are closed and neither being evacuated or refilled (so that isolation valves to vacuum pumps are closed). Probably best if they're filled with Ar (as opposed to under vacuum) to prevent any chance of sucking oil back up into them.
 - Crack inlet tube at KF-25 flange to relieve/bleed vacuum.
 - Turn off pump switch, then unplug from wall.
 - Let pumps cool for a few hours.
 - Set pumps up on a surface (e.g., rolling cart), remove tray (2 screws go through pump, 2 are slotted to let it slide off), and position funnel and waste container below it.
 - Open one or both filling ports for air.
 - Slowly open drain valve and let drain for ~30 minutes.
 - Optional cleaning:
 - Remove sight glass (and cover piece holding it in place), wipe clean, and replace.
 - Rinse/fill pump with cleaning solution.
 - Replace drain plug and fill via either fill port until max fill line in sight glass is reached.
 - Tight fill port caps.
 - Plug in and turn on vacuum pump, then monitor for ~10 minutes to make sure everything's working.

Historical Notes and Pump Settings

- Changed oil in pumps on September 14, 2020 (2020-09-14) for first time since installation (~2.5 years since spring 2018). Replaced oil mist filters on August 18, 2021 (2021-08-18).
 - Upon move into MCMR (late July, 2020), swapped the inlet tubes for the two pumps since the mini antechamber (and hence its pump) gets much more use than the main (large) antechamber.
 - Oil in one pump (for main antechamber?) was still relatively clear (had gone down some, but topped off).
 - Oil in other pump (for mini antechamber/turbo pump roughing?) was noticeably darkened, but not horrible.
 - Drained and changed the oil on both pumps.
 - Noted that both pumps had their mode selector dial turned fully clockwise to the "High Vacuum" setting (as opposed to "High Throughput")
 - The dial on the pump whose oil was still clear was stuck/frozen, so removed the faceplate, tapped the dial with a mallet, and lubricated the threads before resetting to "High Vacuum".
 - Noted that one pump had its gas ballast set to "0" (no gas ballast), while the other was set to "1" (low ballast). The darkened oil was on the latter pump (gas ballast set to "1").
 - The gas ballast adjustment on the pump that was set to "0" was frozen, and we were unable to loosen it, despite tapping with a mallet.
 - After changing the oil, set the gas ballasts on both pumps to "0" since we don't deal with many condensable vapors.
 - This is done by loosening and removing the 3 screws on top of the gas ballast selector, rotating the top plate so that the correct number of divots (0, 1, or 2) aligns with the divot on the lower cylinder, then retightening the screws.