Standard Operating Procedure

Task: Regeneration of the Box Catalyst **Date:** 5/30/2014 Revision Date: 9/18/2017

Background:

 Inert-atmosphere gloveboxes utilize a catalyst bed that traps water using molecular sieves and traps oxygen using a proprietary activated copper material. Over time, moisture, oxygen, and organic solvents saturate the catalyst, reducing its efficiency. Regeneration of the catalyst removes the oxygen and water by heating under a dilute hydrogen stream, ideally restoring the catalyst to its original condition.

Training Requirements:

- Laboratory safety training
- Clearance from glovebox czar, usually after performing a regeneration cycle together

Potential Hazards:

- Compressed Gas Cylinder
- Hydrogen
- Once begun, the regeneration cycle cannot be aborted an insufficient regen gas can damage the box. In case of power outage, the regen restart automatically when the power begins. Sufficient gas must be supplied before the automatic restart.

Materials Needed:

- 5-10% H₂ in N₂ (Regen Gas, Air gas: NI HY10200 or NI HY5200), ~600 kPa, apprx.
 3500 liters according to MBraun
- CGA350 regulator
- Tubing in place, but may need to be reconnected (see below)

Procedure:

- Before beginning the regen, record the time since last regeneration on the last page of the glovebox log.
 - Select the "RKM" figure on the home screen



o Select "Status"



• Record the time since last regeneration:

Purifier		Status				
BACK	EM	END			Alarm	
Automatic active						
Remaining regeneration time :				0	m	in
Time since last regeneration :			9	h	\mathbf{i}	
Total time purifier 1 :			27	h	1	

- Record the details of the regen in the log itself.
- Move the regen gas tank to the appropriate tank clamp location.
 - Poseidon: SG2
 - Hephaestus/Pandora: SG1

- Attach CGA350 regulator (See Changing Tanks SOP for more details).
 - Remember than H₂ regulators are reverse threaded.
- Attach regulator to SG port.
- Attach the tubing from the SG outlet to the regen inlet (green) on the box console and ensure that the regen outlet is inserted into the exhaust line (red).
 - This is particularly important on Heph and Pandora because they draw from the same SG1 line.



- Ensure that the regulator needle valve and all SG outlets are closed
- For glove boxes with lots of solvent use:
 - During Regen fluid from the catalyst will be removed and blow through the flow meter. This corrosive mix has the potential to clog or corrode the flow meter. To avoid problems, a trap must be connected in line
 - Remove the bottom panel on the front side of the regen housing
 - \circ Disconnect tubing from the bottom side of the flow meter
 - o Connect the line to a trap cooled with ice water
 - \circ $\,$ Connect the other side of the trap to the bottom of the flow meter $\,$
 - This connection ensures liquids removed during regen flow from the catalyst, into the trap, and the remaining gas then flows through the flow meter
- Open main valve of cylinder to regulator, then close the valve to leak test. Wait 5 min to see if pressure drops indicating a leak.
 - Ensure that there is enough pressure in the tank for a regen (~600 kPa). A tank has typically been good for two regens.
- Open main valve of cylinder, adjust regulator dial to desired pressure, and open needle valve. Adjust delivery pressure to ~5 psi. Close main valve of cylinder to leak test this part of the system. Wait and monitor pressure.
- Open main cylinder valve, needle valve, and SG outlet. Close main valve of cylinder to leak test full system. Wait and monitor pressure.
- Turn off the Circulation Purifier.

The Miller Group

• Select "Regeneration Purifier"



- First, you must adjust the flow of gas.
 - After the regen cycle has been selected, a warning will appear:



- On the bottom right of the console, there is a flow meter. Changing either the tank needle valve or the SG outlet, adjust the flow to 15 Nl/min (normal liters / min: the amount of gas going through a pipe adjusted to 0 °C and 1 atm). The ball bearing should be between the arrows.
- This step is easier with two people.
- Once the flow is set, click the above button.
- There are 17 steps in a regen. Most of them are proprietary:



- You can check the length of time left of the regen by clicking the status button.
- Once the regen is completed, change the pump oil, purge down the O₂ (~15-20 min), and turn the Circulation Purifier back on.