

Section 5.22 Title: Glovebox Maintenance
Prepared By: Sungho Park and Michael Roy

Revision Date: 11/01/19
P.I.: Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
 Carcinogen Reproductive Toxin High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:
Maintenance of the gloveboxes.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*
6319 ("new" double glovebox), 6325 ("old" glovebox), 6375 ("oldest" glovebox)

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Copper catalyst	Inhalation hazard
Molecular sieves	Inhalation hazard
Activated charcoal	Inhalation hazard

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

High pressure gas cylinders (see gas cylinder SOP)

Exposure Controls: *(Check all that apply)*

PPE: Safety Glasses Face Shield Chemical Splash Goggles
 Chemical Apron Gloves (Nitrile) Lab Coat
 Respirator (type) Other: Long sleeves and fabric gloves; Dust mask
when replacing charcoal or catalyst bed

Engineering Controls:

Fume Hood Biosafety Cabinet Glove box
 Vented gas cabinet Other:

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

Receive training before performing maintenance. Gas cylinders must always be secured with a chain and belt, both tightened around the cylinder circumference.

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Spent catalyst and charcoal are disposed of as solid waste.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Dry spills should be cleaned with a broom and collected as solid waste.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

No formal training or documentation is required. This procedure should be demonstrated by experienced lab members. New members should talk through their procedures with experienced lab members.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry

Signature: _____

Date: _____

Glovebox Maintenance

General guidelines

The glovebox cannot be used for other operations during maintenance and therefore the group needs to be notified in advance. Record in the log book for maintenance operations as well.

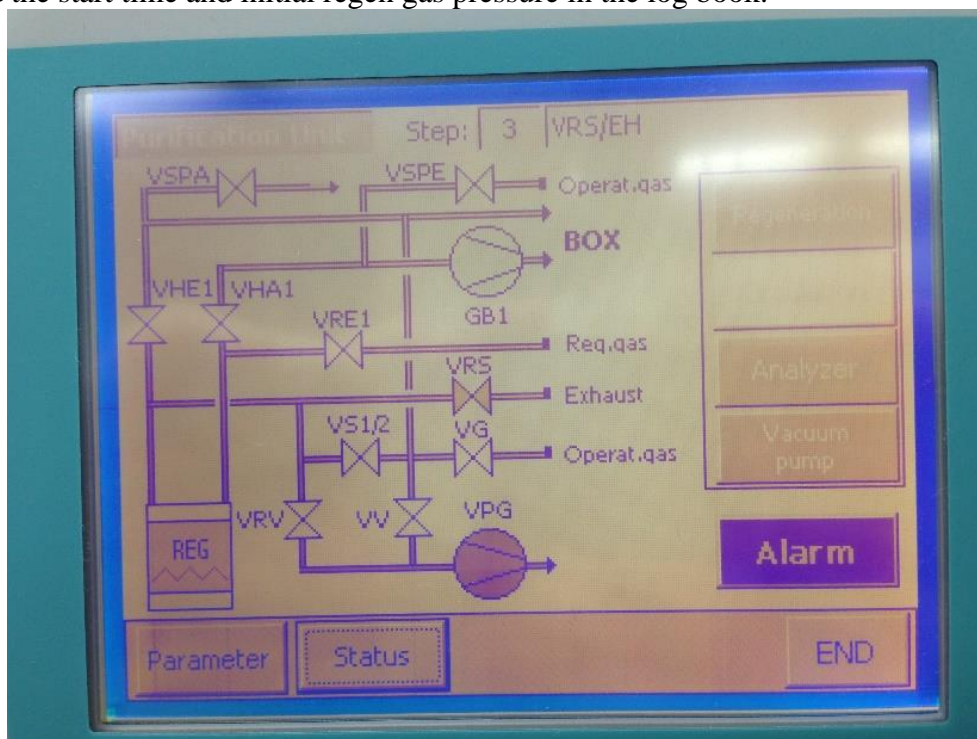
Regenerating the catalyst

If the circulation purifier fails to maintain the glovebox atmosphere (H_2O and O_2 levels < 0.1 ppm), the catalyst needs to be regenerated.

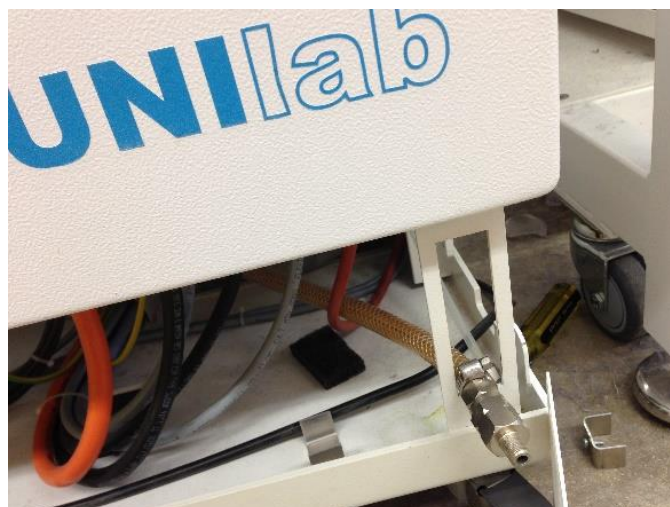


1. Secure the 4% H_2/N_2 tank.
 - a. If the tank does not already have a regulator attached, remove the cylinder cap and place the cap beside the cylinder before installing the regulator.
 - b. There should always be one spare tank reserved on the first floor. Therefore, you need to order another one if you take the one from downstairs.
 - i. Bt 4% Hydrogen Balance Nitrogen Size 300 Batch Analyzed-spec CGA 350
 - ii. Airgas North Central, item code X02NI96B3002168, \$102.19
 - c. Attach the thick hose (tygon tube ending) found on the ground at the back of the glovebox to the 4% H_2/N_2 tank and secure the tubing with copper wire.
 - d. Open the cylinder valve fully and turn back $\frac{1}{2}$ turn. Make sure enough gas is in the tank (> 1300 psi on the high-pressure dial).
 - e. Verify that the low-pressure dial is reading 10 psi.
 - f. Fully open the small valve pointing downwards in the picture above and turn back $\frac{1}{2}$ turn.

2. Turn off the circulation purifier. Connect the exhaust hose found at the back of the glovebox with an extension Tygon tube (placed at the bottom of the glovebox) to enable the exhaust gas to escape through a nearby fume hood. Place a foldable ladder (located in 6319) above this apparatus to prevent people from tripping over.
3. Select the “regeneration purifier” on the touchscreen and select “yes.” The glovebox will allow you to check if the regeneration gas pressure is sufficient prior to the actual regeneration process (you will hear a sound of gas flow). Look at the low-pressure gauge on the regulator and aim for around 8 psi with active flow.
4. Record the start time and initial regen gas pressure in the log book.



- a.
 - i. There is no regen gas flow during the first few steps of the regeneration process. The screen above will be displayed for the first ~3 hours of the regen process with no noticeable/interesting events observable.
 - b. When the regen is complete (~16 hrs, after which the box goes back to circulation mode without any notification), close the small regulator valve and the cylinder valve (leave middle valve as is) and detach the thick hose (look away when detaching the thick hose).
 - c. Typically 900 psi is consumed with 8 psi flow. Record in the log book how much regen gas was actually consumed.
5. In order to prevent rust caused by water that has been removed from the catalyst, detach the bottom lid from the large steel box (where the catalyst bed is located) and blow through the pressure gauge, which contains a small metal ball inside, with air for one hour to remove condensed water in the gauge. Do not use solvent to rinse the gauge.



a. The image above displays the large steel box with the bottom lid removed.

6. Change the vacuum pump oil.

Replacing the N₂ tank

1. Replace when < 300 psi remains in the N₂ tank.
2. Close the cylinder valve and then close the small valve (pointing downwards in the first picture). While looking away, remove the regulator from the cylinder.
3. Obtain fresh N₂ tank (black) from the 1st floor.
4. Attach the regulator. Open the cylinder valve fully and turn it back ½ turn. (again, look away when opening the cylinder valve).
5. Fully open the small valve pointing downwards in the first picture and turn it back ½ turn.
6. Make sure the pressure is regulated at < 60 psi at the low-gauge regulator and purge for ~ 30 minutes.

Replacing the charcoal (located far right, at the back)

The charcoal absorbs some (not all) organic molecules in the glovebox atmosphere. It needs to be replaced after each regen or every year (whichever is more frequent).

1. Detach the cylinder containing the charcoal and bring it out of the box.
2. Empty out the old carbon and replace with new carbon.
3. After wiping the outer surface of the cylinder with isopropanol, put the cylinder in the large antechamber and pull vacuum overnight prior to installation on the outlet port inside the box. The circulator should be off and the glovebox not in use for this overnight period.



Replacing the Hepa filter (located far left, at the back)

The Hepa filter absorbs some (not all) particulates in the glovebox atmosphere. It needs to be replaced after each regen or every year (whichever is more frequent). The Hepa filter involves installation by simply screwing clockwise onto the port located at the leftmost corner of the glovebox.



Order from MBraun (item code 9004513)

Filter Fine HEPA MB-BF-L-03 KF40 DIN/EN 1822 (\$82.40)

Replacing the catalyst

This is caused when the catalyst is irreversibly damaged and regeneration does not restore catalyst ability. Switch off the glovebox and detach from the electrical power supply the giant stainless steel box (picture found above) housing the catalyst.

1. The cylinder containing the catalyst can be unscrewed from the apparatus using monkey wrenches.
2. Empty out the existing catalyst material and put it in the solid waste container. Refill with new catalyst material according to the formula found in #3 and in the order found in #4:
3. Composition: special sieves (5.5 kg) + catalyst (4.5 kg) (order from MBraun)
 - a. Special sieves: Sieve Molecular 13x1/8 APG Pellet 1/8"
 - i. Item code 3240262, \$27.27 per kg, order 5.5 quantities on AESTIVA
 - b. Catalyst: Catalyst Copper Tablet size 5x3mm R3-11(G)
 - i. Item code 2600839, \$102.40 per kg, order 4.5 quantities on AESTIVA
4. Stack 'em up: sieves (2.75 kg) – catalyst (4.5 kg) – sieves (2.75 kg)
5. Reinstall the cylinder containing the new catalyst material using monkey wrenches.
6. Purge the box for 30 minutes.
7. Regenerate the catalyst.

Detaching the vacuum pump (for oil change or for pump repair purposes)

Put both antechambers under static vacuum. Do not detach the oil mist filter from the exhaust it's connected to; instead, disconnect the vacuum pump from the oil mist filter. Move the vacuum pump to a convenient location and change the oil as described in the SOP for vacuum pump maintenance. When the pump has to be repaired and the glovebox is missing a vacuum pump for an extended period of time, the glovebox pressure should be monitored daily and the working pressure range might have to be adjusted since it is now not possible to reduce the glovebox pressure once it goes over the upper limit. The glovebox should not be used when there is no vacuum pump attached.

Replacing gloves

For the old gb,

1. Seal the box with a plug from the inside with the valve facing out the box. Make sure the plastic white parts click on top of the outer metal circumference.
 2. Remove the O-shaped clamp, remove the glove, and determine whether it will be disposed of or used as an emergency glove.
 3. Put a new glove on with the glove O-ring almost completely secured; purge glove with nitrogen flow using a line of nitrogen from the nearby fume hood or countertop tap. Fill the glove, remove the nitrogen line, and slowly roll the glove inwards starting from the fingers. Repeat 10 times.
 4. Remove the nitrogen line and immediately secure the glove O-ring. Put the external O-ring on.
 5. Wrap around the O-ring with electrical tape five times, making sure the tape covers both the glove O-ring and the external O-ring.
 6. Put O-shaped clamp back on so that it covers the glove O-ring but not the external O-ring.
- A similar (simpler) procedure can be used for the new gb. Everything is the same but the black tape and O-shaped clamp are not applicable.

Caution

1. It is important to ensure that the regen gas tank does not run out in the middle of the regen. Hence the flow rate should be strictly monitored to not exceed 8 psi. Occasionally the regulator reading might be “stuck” at a certain value; in this case, try slightly closing and then re-opening the middle valve.
2. Do not rely on the circulation purifier to lower the high O₂ and H₂O levels that you see after use. This can saturate the catalyst more quickly, leading to more frequent regen cycles during which glovebox use is not possible. The circulation purifier exists primarily to maintain the atmosphere while the box is idle; if you want to clean up the glovebox atmosphere, you need to do a quick purge.
3. The analyzer will not be accurate when the circulation purifier is turned off.

Regional Representative (new GB)

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