

Vigor Glove Box

Standard Operating Procedure

Lab: ESB 154

Department: Materials Science and Engineering

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Section 1: Overview

Type of SOP: ☐ Process ☐ Hazardous Material ☐ Hazardous Class of Materials ☒ Equipment

Synopsis:

This SOP is written to standardize the operating procedure of the Vigor glove box, protect the equipment from being used improperly and protect the users from potential hazards.

Section 2: Risk Assessment Summary (Hazards and control measures)

Materials:

| Material (name, CAS #, other ID) | Hazards |
|----------------------------------|---|
| Argon, 7440-37-1 | High pressure, suffocation when leakage happens |

Equipment Hazards:

Bodily Injury: the glove box is connected to a continuous supply of argon gas from a high-pressure liquid argon tank. In the event that the tank is in need of replacement, DO NOT attempt to do this yourself. The tank is heavy and can cause severe physical harm if moved improperly. Please notify Alan Qu and/or Sanghyeon Kim of the need and certain measures will be taken.

Hazardous Conditions:

Reduced/Shut-down of gas supply: circulation will be shut down and materials enclosed are subjected to contamination.
Overpressure of gas supply: equipment is subject to damage and can cause physical harm to human body.

Technique Hazards:

Overtighten big antechamber door: door can be damaged during refilling and can cause physical harm to human body.
Improper use of sharp: can cause glove damage, inhalation of toxic gas, contamination of enclosed materials.

Personal Protective Equipment

Nitrile gloves, Safety glasses, Lab coat, Close-toe shoes

Engineering Controls

The glove box is connected to a protector with an ON/OFF switch at all times (on the sidewall of the control box, right-down side), in case an emergency shut-down is required.

The argon tank has two safety valves in case flow of gas into glove box must be cut off.

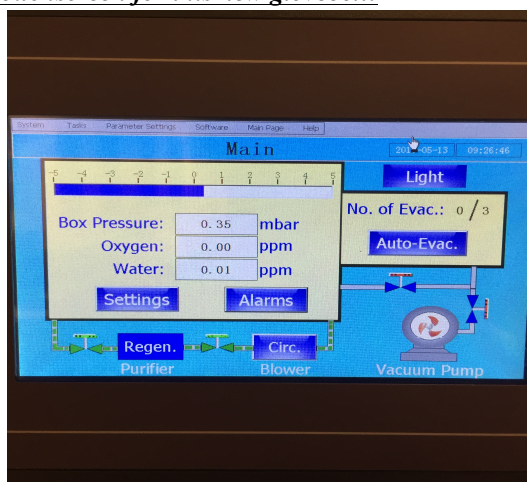
The argon tank has a built-in over pressure outlet that prevents tank from being over-pressurized.

Section 3: Procedures


Sign in the logbook hung right by the glovebox before and after your experiment.

Pay extra attention to the oxygen level, if it is above 5ppm, please notify Alan Qu or Sanghyeon Kim.

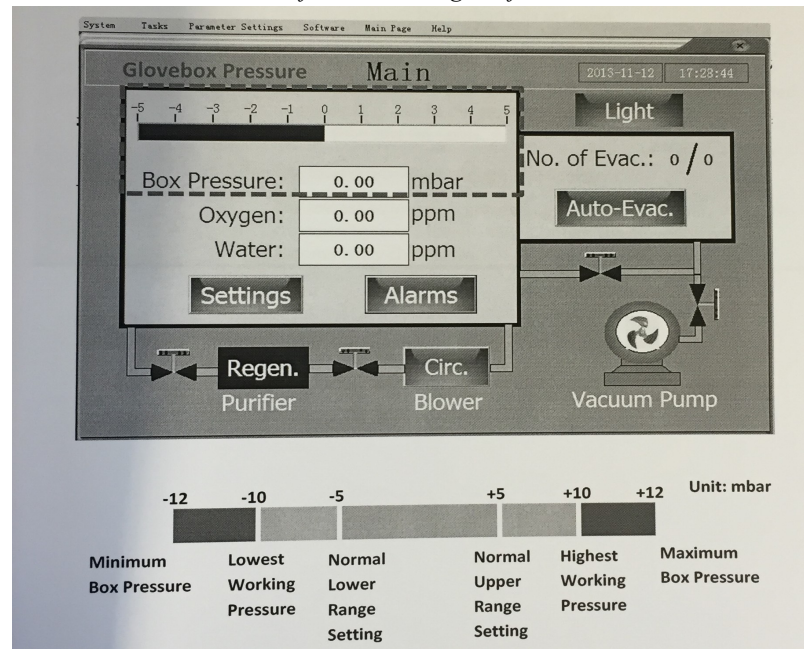
We have a multifunctional touchscreen for this new glovebox:



There are several accessible functions on the main menu page.

| | | |
|---|---------------------------|--|
| Alarms | Alarm Setting | When the system encounters problems, this button will flash. Press the button to enter the Alarm page to get specific information. |
| Auto-Evac. | Large Antechamber Control | Touch this function button to enter the Auto-Evacuation page to set the auto-evacuation parameters and to start auto-evacuation of large antechamber. |
| Circ. | Circulation | Touch this function button to enter the Circulation page to control the purifier circulation. |
| Light | Light | Touch this function key to turn the glovebox light on. Touch again to turn the light off |
| Regen. | Regeneration | Touch this function button to enter the purifier Regeneration page. |
| Settings | Setting | Touch this function key to enter the System Settings page where the system parameters are entered and displayed. The system parameters entered here include the Box Pressure limits and the impurity levels necessary to cause an alarm. |
|  | Vacuum Pump Control | This touchscreen icon represents the vacuum pump. Touching the icon once will turn the vacuum pump on and touching it again will turn it off. |

Glovebox Pressure: The pressure limits set for the glovebox interior are relative pressures, referenced to the surrounding atmospheric pressure. A positive pressure indicates the pressure inside the box is greater than the outside atmospheric pressure while a negative pressure indicates a lower pressure than outside the glovebox. The unit of the pressure used here is the mbar, or one thousandth of a bar. Generally, operating pressures limits between 0 and a few mbar are good for most users.



It is also suggested to read the oxygen level from the GE oxygen sensor placed on top of the glovebox.



Functions operation: When you click the “Task” button on the touch screen there are several operations that you can do to maintain the good working conditions of the glove box. Important ones listed below.

Please remember that Purging and Regeneration should ONLY be done by Sanghyeon Kim, Junjie Wang or Alan Qu. Circulation can be done by any trained user.

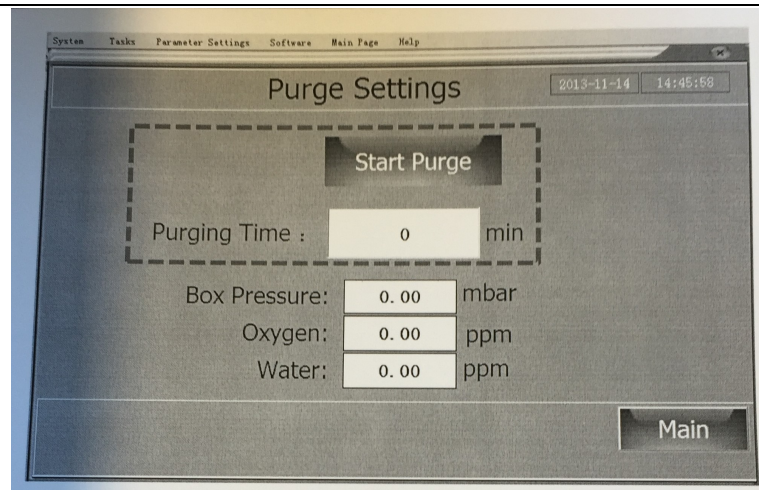
- 1) ***Purge:*** When impurities are introduced into glovebox, we recommend turning off circulation system and purging the impurities out of the box (5-10 minutes). Doing so will avoid introducing the impurities into the gas purification column and prematurely consuming the purification capacity.

Caution!

1. Set the Box Pressure upper limit to 8 mbar and lower limit to 5 mbar before activating the purge function.
2. Be sure that working gas is being delivered at a minimum **60 psi (~4bar)**. If not, the glovebox will be unable to open the pneumatic valve when vents purge gas.

Warning!

To activate the purge function, the Circulation and Regeneration need to be completely turned off. Circulation should be set to manual and then turned off.



Enter a value in the Purging Time box and press Start Purge to activate the purge function. Purging will stop after the specified time period. It can be stopped at any time by pressing the Stop Purge button.

- 2) **Circulation:** After a pure or regeneration cycle completes, the circulation function removes residual impurities to < 1ppm. Note: The very first circulation, performed during set up, may only bring impurities to single digits before regeneration is needed. Thereafter, it will achieve an impurity level of < 1ppm until it is time to regenerate it again.

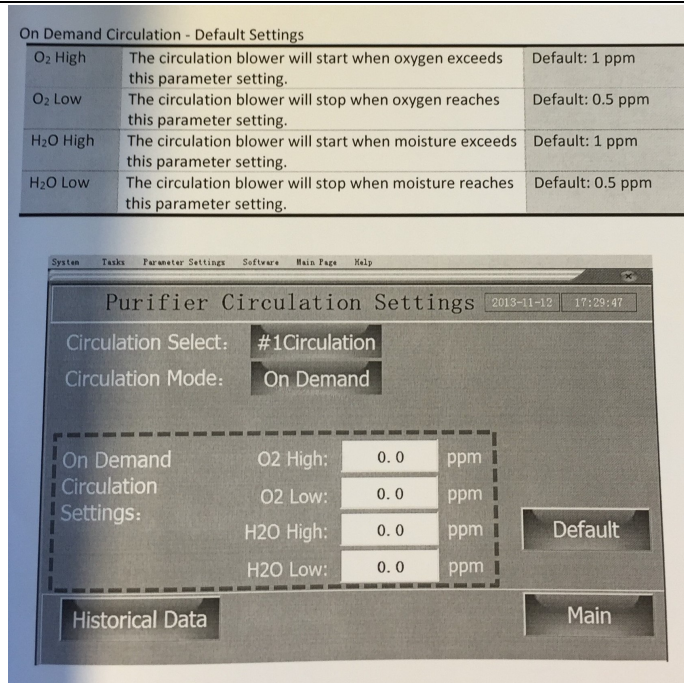
Warning!

Before starting the circulation, please check to assure that:

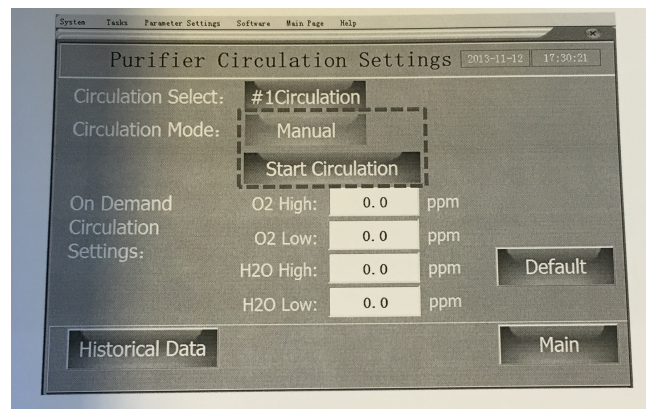
1. The purge function is not running.
2. Any running regeneration cycle has fully completed and that the purifier(s) is/ are completely cooled down to room temperature. Please **DO NOT** restart circulation before the regeneration fully completes (20 hours) as this may damage the glovebox permanently.
3. At least **70 psi (~5bar)** of working gas pressure is available to allow circulation to start. If the pressure is too low, circulation will not start.
4. The oxygen level is < 100ppm (or from 100 – 200 ppm during initial startup).

Press Circ. On the main menu page to enter the circulation page. There you will specify whether you want the purifier to circulate in manual or automatic (on demand) mode.

On Demand Circulation Mode is the default mode for all Vigor gloveboxes. Under On Demand mode, circulation will start when the oxygen or moisture level exceeds the upper limit (which you have previously selected as suitable for your work, see settings window shown below). Circulation will continue until the impurity level reaches the lower limit that you set.



***Manual Circulation:** To control the circulation function manually, press the On Demand button once to transfer into the Manual control mode. A new button, Start Circulation, will appear. Press this once to start manual circulation. It will continue to run until you press the button again (it now appears as Stop Circulation).*

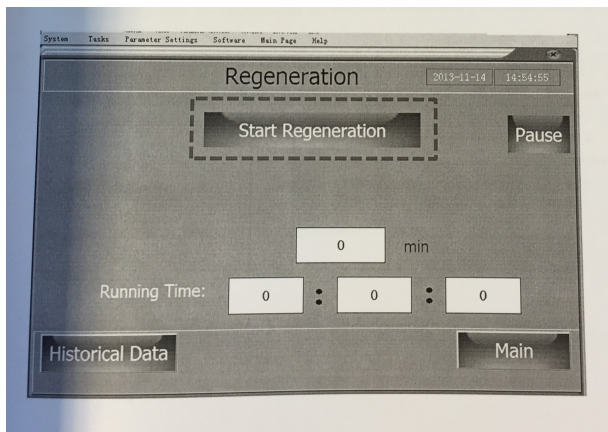


For a new installation, Vigor recommends setting the circulation mode to Manual, and running the circulation continuously for at least one week, or setting it to On Demand mode with the upper limits for O₂ and H₂O set to 1ppm and the lower limits for O₂ and H₂O to 0.2 ppm. During this startup period moisture slowly (over days) desorbs from interior surfaces and moisture gradually drops. Oxygen will drop much faster than water in low ppm ranges.

- 3) **Regeneration:** When running the circulation process becomes unable to remove impurities from the glovebox to reach the selected levels, or when this process greatly slows, regeneration is indicated. To enter the regeneration page, press the Regen button on the main menu.

Warning!

Do not use pure hydrogen gas or regeneration gas with a hydrogen level exceeding 10% for regeneration due to the possibility of ignition or explosion. Vigor suggests using an ultra-high purity grade mixture of 5% Hydrogen in 95% inert gas (to match your working gas), supplied at 15 psi, for regeneration.



After adjusting regeneration gas flow, please click Start Regeneration icon to start regeneration. Regeneration will proceed for the next 20 to 24 hours. To manually turn off the regeneration process, please click Stop Regeneration (Scram button on some models) to stop the process. Although, unless there is a compelling reason why this is necessary, we suggest you not interrupt the process.

Caution!

- 1. After regeneration, impurities levels might have risen by a few ppm which is normal and mostly due to diffusion through the gloves. Starting the circulation will quickly bring the levels of impurities back down again.*
- 2. After regeneration when starting circulation, the gloves might suck into glovebox or deflate all the sudden due to the vacuum state of the purifier, which is normal.*

Before-experiment inspection: *Check the H₂O level on the panel, DO NOT use if it is higher than 10ppm. Check the gauges on the argon tank, make sure there is enough gas and the pressure on the left gauge is right above 80psi (a little bit higher than 80psi).*

Loading samples: *Make sure the insides doors are closed before loading samples.*

- i) if using the small antechamber, the chamber should be pumped and refilled three times before loading samples into the glove box.*
- ii) if using the big antechamber, the chamber should be pumped ONLY once for at least 30 mins and refilled with argon before loading samples into the glove box.*

Use two fingers to close the big antechamber doors. DO NOT overtighten the big antechamber doors.

Restart circulation and analyzer: In some cases, e.g. the gloves are pushed too fast, the circulation and/or analyzer will be shut down. When this happens, press “Task” on the panel and select “circulation” in the slab, type in the upper and lower limit of desired H₂O and O₂, the circulation will be turned on.

Equipment error information: In some cases, the panel will show “inlet/outlet valves purifier not opened”, first check the gauge of tank, normally it is due to the argon gas is running out. Replace the gas tank and restart circulation and analyzer if necessary (refer to above). If there is still gas inside the tank and the error info is not gone, try to increase the pressure above 80psi a little and when the valves are back on, REMEMBER to reduce the pressure back to 80psi. If this still doesn't work, contact the safety person.

Section 4: Waste Disposal/Cleanup

The waste are to be disposed of according to Division of Research Safety (DRS) ChemTrek requirements. Clean up after experiment. Dispose of any wasted labwares (gloves, pipettes, kimwipes, weighing papers, etc.) OUT of the glove box.

Section 5: Spill/ Other Emergency

1. Please make sure a piece of aluminum foil is spread out on the bottom inside the glovebox when you are handling powders, and please wrap up and dispose of it properly according to DRS requirements.
2. When there will be volatile chemicals involved in your experiments, please make sure you seal the containers once finished so as to prevent potential damage to the catalyst and regenerator.
3. If some liquid chemical (flammable) is spilled, please use kimwipes to clean it up and take the kimwipes out of the glovebox after operation.
4. According to DRS spill control stipulations, please take certain measures on corresponding chemicals and notify Alan Qu and/or Sanghyeon Kim. If it is something immediate out of your control by your sensible judgement, call the DRS or 911.

Section 6: Additional Information

Advice:

1. Usage of water or any aqueous materials is NEVER allowed inside glovebox.
2. Squeeze bottles with liquid inside are NEVER allowed inside the antechamber during pumping down.
3. Degassing of porous materials (e.g. kimwipes) is necessary for at least 30 mins before opening antechamber from inside (overnight for a whole box of kimwipes and gloves, too!).
4. While refilling the big antechamber, DO NOT refill argon too fast, open the refill valve to the half at first, once the pressure reaches to -0.4, the refill valve can be fully opened.

Checklist:

A checklist can be written in the SOP as a reminder for the steps needed to take in order to perform the procedure. Potential checklist items include:

☒ ***Read (Material) Safety Data Sheets.***

☐ *Proper fire extinguisher is nearby.*

☐ *Another researcher is nearby and knows the hazards present.*

☐ *All calculations are done prior to beginning the procedure.*

☐ *The required glassware is of the proper size to accommodate all steps of the procedure.*

☐ *Received necessary immunizations.*

[illegible]