University of Minnesota Nano Center Standard Operating Procedure

Equipment Name: Controlled Atmosphere Glove Box
Model: Labconco Protector
Location: PAN 185
Revision Number: 3
Revisionist: J. Marti
Last Revision Date: 1/3/2019

A. Introduction

1. **Tool Description.** The Labconco Protector Controlled Atmosphere Glove Box allows materials to be manipulated while isolated from oxygen and moisture. Starting with ambient air, the glove box uses a vacuum pump to purge its volume, and then fills the glove box with dry nitrogen gas form a cylinder. This process is repeated automatically for a programmed number of cycles. A level of <4000 ppm of O_2 can be reached after about 40 purge-fill cycles, after which the gas purifier (see below) may be used to bring the O_2 and H_2O levels down to 1 ppm.

   The glove box will typically be left filled with an atmosphere with oxygen concentration at or below 5000ppm, so minimal pump down time should be required. Materials and tools may be introduced into the box using a load-lock, which removes the need to vent the box when adding or retrieving items.

   The system is equipped with an AtmosPure gas purifier that recirculates the glove box air and scavenges oxygen and water vapor to as low as 1 ppm. This system is engaged ONLY after oxygen and water levels in the box have reached 4000 ppm or below, using the automated purge/fill cycle of the main glove box.

2. **Tool Components**. Figure 1 shows a front view of the glove box. Key components visible in this view are
   a. Glove box body. The box is a stainless steel-lined single width chamber with two glove ports, large viewing window, and internal lighting. The chamber contains an AC power outlet and an internal door which opens to the load port.
   b. Transfer chamber. This load chamber can be vented and pumped down independently of the main chamber, so that equipment and tools may be passed into the main chamber without venting it.
   c. Vacuum pump
   d. Nitrogen cylinder
   e. AtmosPure Re-Gen gas purifier. This system recirculates the glove box atmosphere and scrubs oxygen and water from the gas. Using the Re-Gen purifier, it is possible to attain oxygen and H_2O concentrations down to 1 ppm.
   f. Oxygen monitor
   g. Water monitor

3. **Control panel**. Figure 2 shows the front control panel menu to manage glove box operation. The panel display toggles between glove box pressure control, glove box purge and fill, and transfer chamber purge. The menus are selected using the upper right mode key on the front

control panel. The function of these panels is discussed below.

Figure 1. Key components of the controlled atmosphere glove box.

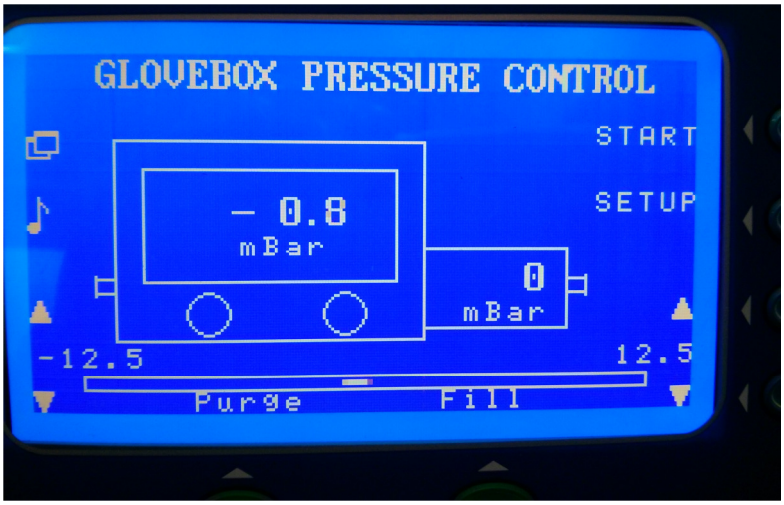

Figure 2. Control panel display. This display toggles between glove box pressure control, glove box purge/fill, and transfer chamber purge/fill.

4. **Safety**. Although the glove box has been engineered to maintain optimum operator safety, caution should always be used while working. Prior to using the glove box, check to make sure that the main chamber pressure is set for comfortable glove manipulation. Changes in glove position will affect pressure in the glove box.

5. **Restrictions/requirements.** Before using the glove box, provide the lab manager with the names of any chemicals you plan to use in the box, along with their safety data sheets. **All chemicals must be approved by the lab manager before they are brought into the glove box.**
    a. The glove box is not explosion proof. The use of flammable gases or solvents in the glove box should be limited. Users must determine the lower explosive limits and flammability of the gases used within the controlled atmosphere glove box, and ensure that gas or vapor concentrations stay under the LE. If in doubt, consult with MNC staff.
    b. Do not expose the oxygen monitor to fumes of organic solvents (alcohols or nonpolar solvents). If using these solvents, set the sampling line valve to room air (see below).
    c. Do not use concentrated acids or bases inside the glovebox without approval of the lab supervisor. Perchloric acid use in this glove box is prohibited.
    d. Contact the lab manager if you plan to use hazardous materials in the glove box. Special venting connections will need to be in place prior to starting your project.

    Use good housekeeping in the glove box at all times. If you will be transferring liquids or powders inside the box, place Kimwipes or lab pads on your work surfaces to absorb any spills, and load extra wipes along with your materials at the start of your session. Remove these pads when done and discard. Do not use water or solvents to clean any surfaces. Contact the lab manager in the event of a major spill.

6. **Required Facilities.** 120V AC power, dry nitrogen gas.

7. **Definitions**
    a. Purge: pump-out of air inside the glove box, using the vacuum pump. The system purges the chamber down to a pressure 12.5 mbar (5" water) below atmospheric pressure.
    b. Fill: back-filling the purged glove box volume with dry nitrogen gas from the cylinder. The system fills the chamber to a pressure 12.5 mbar (5" water) above atmospheric pressure.
    c. Ambient/inert atmospheres: ambient atmosphere (room air) contains about 20% O_2 by volume. An inert atmosphere may be defined as one where oxygen content has fallen below some critical threshold. For our purposes, an inert atmosphere is one having oxygen concentration at or below 0.4%, or 4000 ppm.
    d. ppm: parts per million.

B. Using the Glove Box

1. **Set Up**
    a. Begin a new entry on the Glove Box log sheet. List your name, the date, the material you are working with inside the box, the final O_2 and water concentrations

measured at the end of your run (in ppm), and any issues you encountered during your run.

b. Turn on the oxygen and water vapor monitors if not on already; the O_2 monitor requires a warm up of about 5 minutes.

c. When ready to start reading levels in the chamber, locate the sampling valve behind the monitor and turn it to allow flow from the glovebox into the gas monitors. After you are finished with the glove box return the valve to the room air position.

d. Open the house nitrogen valve on the wall behind the glove box.

e. The box will usually be left with a partially inert atmosphere, that is, one having about 4000 - 5000 ppm (0.4 to 0.5 %) of oxygen or water. If you need oxygen or water levels lower than this, you will need to do some purge/fill cycles to reduce these gas concentrations--see item 3 below.

2. **Loading the Glove Box.** To preserve the atmosphere inside the glove box, the transfer chamber is used to load items from the outside environment.

a. Check to ensure that the inner transfer chamber door is closed and sealed. If not, close it and lock it with the locking handle.

b. Check the position of the four manual valves on the front of the transfer chamber. These valves are shown in Figure 3. Move all valves to the "open" position if not already open.

c. Toggle the front panel display to bring up the transfer chamber pressure screen. The chamber should be at or near atmospheric pressure, given by a value of 0 +/- 3 mbar. If the chamber is under-pressured, use the "fill" key on the transfer chamber purge/fill menu to bring the pressure to ambient (i.e., a value of "0").

d. Open the outer transfer chamber door and load your tools/materials, then close and lock the outer transfer chamber door.

e. Use the front selector panel to bring up the transfer chamber purge/fill menu. The number of purge/fill cycles (to replace the air in the chamber with dry nitrogen) should be set to 1; more than one purge/fill cycle is not necessary.

f. Press "Start" to begin purging.

g. After the purge/fill cycle is complete, the inner transfer chamber door may be opened by reaching into the glove box. Retrieve and position your equipment and materials.

h. Close and lock the inner transfer door.

i. Check the oxygen and water meters. The act of loading may have raised both oxygen and water vapor levels. Depending on your requirements, 1-2 additional purge/fill cycles for the main chamber may be necessary to reduce these gases.

3. **Main Chamber purge/fill (pump down) sequence.** To decrease the oxygen or water vapor levels in the box, do the following steps.

a. Check the position of the four manual valves on the front of the transfer chamber. These valves are shown in Figure 3. Move all valves to the "open" position if not already open.

b. Toggle the front panel display to bring up the glove box purge/fill screen.

Figure 3. Manual valves on the front of the transfer chamber.

c. During a purge/fill cycle, the system will automatically pump the box down to a pressure of 12.5 millibar (mbar) below ambient, then fill the chamber with dry nitrogen up to a pressure of 12.5 mbar above ambient. This process is repeated for as many cycles as the user selects in the glove box purge/fill screen (1 to 199 cycles). Figure 4 shows a graph of the expected level of oxygen attained after a given number of purge/fill cycles.

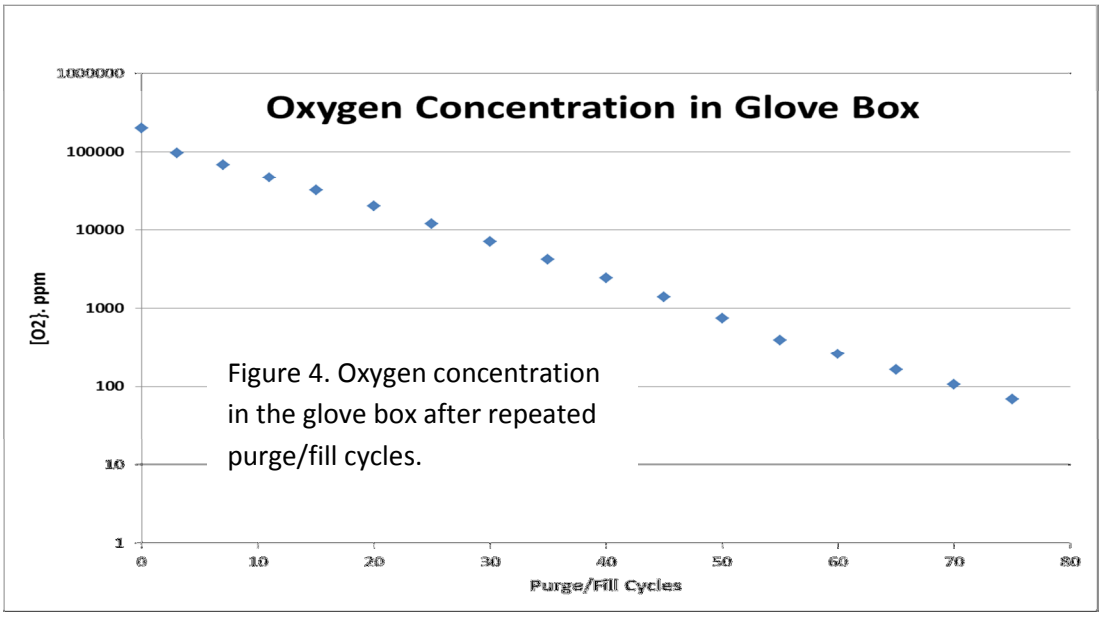

Figure 4. Oxygen concentration in the glove box after repeated purge/fill cycles.

d. Consult Figure 4 to help select the number of purge/fill cycles that will bring the O_2 concentration down to 4000 ppm if it is not already at this level. Starting from atmospheric conditions (20% oxygen), about 40 cycles will be needed, which takes about 40 minutes. Since the glovebox is usually kept at 4000 to 5000 ppm O_2, fewer cycles should be needed.

e. After the purge/fill cycles have completed, verify that the levels of O_2 and H_2O are 4000 ppm or lower. If not, run through 5-10 additional cycles.

f. If you do not require levels lower than 4000 ppm, you may begin your work in the box. If lower levels are desired, go to "Using the Gas Purifier" below.

NOTE: At the end of the purge/fill cycles, the glove box is usually over-pressurized, and the gloves are fully inflated. This makes it impossible to insert your hands into the gloves. Use the "purge" key on the glove box pressure control panel or the footpad to drop the pressure and pull the gloves inside the box (a pressure of about -0.5 mBar). Adjust the pressure if needed during your session to ensure a comfortable position of the gloves.

4. **Using the Gas Purifier.** Very low levels of O_2 and H_2O (down to 1 ppm) can be reached using the AtmosPure gas purifier, which circulates the glove box atmosphere through a molecular sieve (to scrub water) and a chemical oxidation cell (to consume O_2). The gas purifier is to be used ONLY after the levels of O_2 and H_2O have dropped to 4000 ppm or below.

   The front panel control is shown Figure 5. To engage the purifier:
   a. Turn on the main front panel switch on the AtmosPure.
   b. Switch the blower mode to "on".
   c. Open both manual valves located on the right side of the unit.
   d. Monitor O_2 and H_2O levels, which should start dropping soon after starting the purifier.

Figure 5. Gas purifier front panel.

5. **Glove box stability.** The leak rate for the glove box is quite low. As shown in in Figure 6, the oxygen permeation rate is 0.30ppm/min. Depending on the length of your session, you may require additional purge/fills or use of the gas purifier to maintain your desired O_2 and H_2O content.

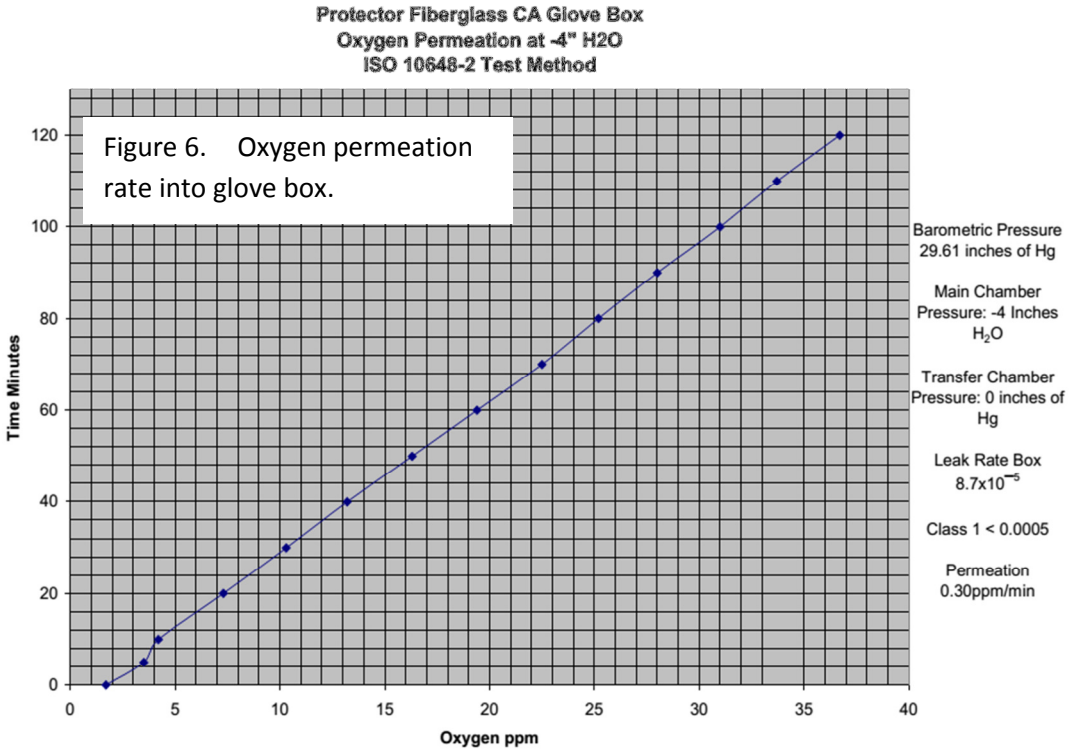

Figure 6. Oxygen permeation rate into glove box.

6. **Glove box Shutdown.** When your work is complete:
   - **a.** If using the gas purifier, turn it off and close the manual valves on the right side of the purifier box.
   - **b.** Check the transfer chamber pressure to ensure that it is close to that of the glove box (they should both read close to atmospheric, i.e., near zero over- or underpressure).
   - **c.** Open the inner transfer chamber door and move all your materials to the transfer chamber, along with clean up wipes and any equipment not approved by MNC staff to remain in the glove box. Ensure that the chamber surface has been cleaned of any material spills.
   - **d.** Close and seal the inner chamber door.
   - **e.** Retrieve your items by opening the outer transfer chamber door. Close and seal the door when done.
   - **f.** Note the O_2 and H_2O levels on the log sheet for the next user.
   - **g.** Switch the valve on the gas monitoring line to sample room air. Leave the O_2 and H_2O monitors on.