Glove Box Procedures

Cameron Dale
August 18, 1999

This report is a detailing of the steps required to operate normally, and to regenerate, the in-house built glove box. The glove box was constructed by Ou Mao and Frank Van Gils, and if there are any questions regarding the use of the box, they should be directed to Frank. Note that to understand and use these steps there are three additional pages necessary, which should be attached to this document or available next to the glove box. The first is a picture of the glove box system, with all the valves and gauges labelled numerically. The second is a table of the conversion from emf in millivolts to relative temperature in °C of a type K thermocouple. The third is a calibration of the lifetimes of filaments as a function of O_2 and H_2O contamination. There is also a sheet somewhere near the glovebox for recording the usage of it. Information should be recorded on it whenever the environment is tested or a regeneration is done.

1. Normal Operation

This section deals with the normal day-to-day operation of the glove box. This involves many things, from inserting and removing objects to or from the glove box, to testing it's contamination level.

1.1. Using the Glove Box

The inert Argon environment of the glove box is continuously circulated through the gas purifier to remove any unwanted gases from it. The blower seen on the attached diagram continuously circulates the air through the purifier. Normally, the only open valves should be 1 and 2 to allow this circulation, and possibly 3 which allows gauge 14 to measure the pressure in the purifier.

The glove box is kept over-pressured with Ar to minimize the amount of contamination. This over-pressurization can be checked on gauge number 16, which shows the pressurization in inches of water above atmospheric pressure (1 atm = 407.1" H_2O). In the normal use of the glove box, such as inserting your arms into the gloves, the pressure inside can change by large amounts. There are two outlets from the glove box, labelled 19 on the diagram, which allow the air to escape through some oil if the pressure exceeds 1.5" H_2O. This means that when the glove box is returned to its idle state, the pressure will be lower and may even be

below atmospheric pressure. This can be remedied by switching valve 10 up to the glove box side, and opening flow controller 12 to allow more Ar to enter the box.

1.2. Inserting and Removing Objects

Inserting and removing objects is done through either of the two cylinders located to the left of the glove box. If at all possible, the smaller cylinder should be used for this rather than the large, as the pumping times will then be shorter and the risk of contamination smaller. The basic procedure is to pump the cylinder to vacuum, then flush it with Ar, and then repeat until the cylinder is reasonably free off contamination. The actual procedure for doing this for each cylinder is described below. Once this has been done, the cylinder door inside the glove box can be opened, and any objects inside it can be taken into the glove box. To remove an object is now easy, as the object can be placed in the cylinder, the cylinder door inside the glove box closed, and then the outside door opened and the object removed.

Below is the procedure to insert an object into the glove box using the small cylinder.

1. Make sure the cylinder is not at vaccuum by opening valve 9 up to the glove box, and then returning it to the closed (horizontal) position.
2. Open the outside door of the cylinder, place the object(s) in the cylinder and firmly close the outside door. Ensure that the object(s) have no sealed pockets of air that will contaminate the interior of the glove box.
3. Pump out the cylinder by turning on the pump, and turning the valve 9 down to the pump direction.
4. After a suitable amount of time (5–10 minutes), turn valve 9 back to the closed position.
5. Slowly turn valve 9 up to the glove box to allow air from the glove box to enter the cylinder.
6. Once the cylinder is at the same pressure as the glove box, turn valve 9 back to the closed position.
7. Repeat steps 3–6 two or three times to insure the cylinder is clean. If shorter pumping times are used, then the number of repetitions should be increased. Also, watch gauge 16 to make sure that the glove box does not become under-pressured.

Below is the procedure to insert an object into the glove box using the large cylinder.

1. Make sure the cylinder is near atmospheric pressure by checking gauge 15. This gauge measures pressure in inches of Mercury (1 atm = 29.92" Hg). Note however that it measures this relative to atmospheric pressure, and so a reading of 0" Hg is atmospheric pressure, whereas a reading of 30" Hg is vaccuum.

2. Open the outside door of the cylinder, place the object(s) in the cylinder and firmly close the outside door. Ensure that the object(s) have no sealed pockets of air that will contaminate the interior of the glove box.

3. Pump out the cylinder by turning on the pump, and opening valve 7.

4. After a suitable amount of time (15–20 minutes), close valve 7.

5. Admit Ar to the cylinder by slowly turning valve 10 towards the large cylinder.

6. Once the cylinder reaches atmospheric pressure, according to gauge 15, close valve 10.

7. Repeat steps 3–6 two or three times to insure the cylinder is clean. If shorter pumping times are used, then the number of repetitions should be increased.

1.3. Testing the Environment

Testing the environment in the glove box is done using exposed lightbulb filaments inside the glove box. The lifetime of the filament in the environment as a function of the amount of O_2 and H_2O present is linear when plotted on a log-log scale. A rough idea of the amount of contamination (in parts per million) can be obtained from the attached calibration graph. This graph is for 40W G.E. light bulbs, which should be available in the lab.

Below is the procedure for testing the environment of the glove box for contamination. Note that the specially designed lifetime timer consisting of a switch, dimmer, clock, and counter, should be available in the lab on top of the glove box. This device is connected to a lightbulb socket located in the glove box, normally on a shelf on the right hand side.

1. Insert a 40W G.E. lightbulb and a carbide tipped scriber (or other glass cutting tool) into the glove box using the procedure outlined in the previous section.

2. Inside the glove box, scratch the end of the light bulb in a circle with the carbide tip. Considerable force is required to do this, however be careful not to puncture the gloves.

3. Inside a ziploc bag, carefully strike the scratched circle with a blunt object until the lightbulb breaks open. If the filament breaks in the process of this, then the lightbulb is ruined and the procedure will have to be restarted. It is a good idea to bring two or three lightbulbs into the glove box in case this occurs. Make sure that all the broken glass is collected in the bag, and not left in the glovebox.

4. Screw the lightbulb with the exposed filament into the socket in the glove box.

5. Turn the lifetime timer on, and turn the dimmer up all the way. The filament should light and the timer begin to count. If this does not occur, insure that the filament is not broken and that the switch on the socket inside the glove box is on.

6. When the lightbulb filament breaks, the timer should stop. The lifetime of the filament can then be used with the attached graph to determine the approximate concentrations of impurities in the glove box.

2. Regenerating

Next to the glove box is a gas purifier which the inert gas in the glove box is continually circulated through. It consists of a screen, two molecular sieves, and some oxygen remover. Over time, the small amounts of oxygen filtered from the system build up in the oxygen remover. This regenerating process is intended to flush this material of oxygen, so that it may return to a high efficiency of oxygen removal. This regeneration should be done as required to keep the environment in the glove box reasonably free of impurities. Concentrations as low as 1 ppm are possible with the current system.

The basic idea in the regenerating process is to first isolate the gas purifier from the glove box, and pump it to a rough vaccuum. Then, Helium gas is added and the gas purifier is heated to a temperature between 130°C and 250°C. The purifier is then flushed with a mixture of Argon and Hydrogen to remove the built up oxygen. It is then pumped out and allowed to cool. The following steps will illustrate this process in great detail. At the beginning of this process, all the valves should be closed except for 1, 2, and 3.

1. Connect the two thermocouples to DMMs: thermocouple 20 is the one on the right, 21 is on the left. Also connect the heating cable to the Variac, though make sure it is not on yet.

2. Close valves 1 and 2 to isolate the purifier from the glove box.

3. Turn on the pump and open valve 6, then slowly open valve 5. Gauge 14 should begin to change from 0" Hg to 30" Hg. Note that this is a measure of the pressure relative to atmospheric, as is gauge 15, and so 30" Hg is vaccuum.

4. After a few minutes, the purifier should be at a rough vaccuum. Close valve 5.

5. Disconnect the tank of Ar and H_2 from valve 4, and connect a tank of He gas. As the connection is being made, the He should be flowing slightly out of the hose, to remove any unwanted gases in the hose and connection.

6. Open the flow controller on the He bottle and valve 4 slightly to admit He slowly into the purifier. At about $\frac{2}{3}$ atmospheric pressure (10" Hg) stop by closing valve 4 and the He flow controller.

7. Apply 80–90 V to the heater by the dial on the Variac. Note that this dial reads from 0 to 100, which is not volts. At the present time, a setting of 60 gives approximately 85 V, but this can be checked.

8. The thermocouples which should previously have read almost no voltage should now begin to rise. Note that these measure temperature relative to a reference, which in this case is room temperature (25°C). Therefore, the temperature can be found on the attached conversion sheet, and should then have 25°C added to it to get the actual temperature.

9. Heat the purifier until the temperature of one of the thermocouples reaches 250°C. In this heating, neither thermocouple should exceed this temperature. Also, be careful to monitor gauge 14, as the pressure should not exceed atmospheric pressure (0" Hg). If it approaches atmospheric pressure, open slightly valve 5 (with the pump still running) to allow some air to escape.

10. When one of the thermocouples reaches 250°C (in about 3 hours), unplug the heater from the Variac.

11. Wait for the two thermocouples to become more homogeneous, when they are less than a millivolt apart is good enough (about an hour). In doing this, neither thermocouple should drop below 130°C, and if one does then the heater should be plugged in to try again.

12. Disconnect the He from valve 4, and reconnect the Ar and 5% H_2. In doing this, the gas should again be flowing as the connection is made to blow out any impurities.

13. Close valve 6 and turn off the pump.

14. Disconnect the hose between valves 5 and 6, and use it to connect valve 5 to the pump exhaust through the T-connector labelled 11.

15. Open flow meter 13 slightly on the Ar mixture, and open valve 4 to let it flow into the purifier. When the pressure on gauge 14 is above atmospheric, open valve 5 slightly to allow the gas to escape.

16. Adjust flow meter 13 and valve 5 until the Ar mixture flows through the system and the flow meter reads about 50.

17. After a while of this flowing (5–10 minutes), thermocouple 20 should begin to heat up. Then, after about another 5–10 minutes it should cool down again. If it approaches 250°C in this heating, turn down the flow rate by adjusting flowmeter 13.

18. After about another 20–30 minutes of flushing, thermocouple 21 should heat up and then cool down. Continue flushing for another 5–10 minutes.

19. Close valves 4 and 5 and flow controller 13.

20. Disconnect the hose from T-connector 11 and reconnect it to valve 6.

21. Turn on the pump and open valves 6 and 5 to pump out the gas purifier.

22. Continue pumping until the thermocouples are close to 50°C (about 24 hours).

23. Close valves 5 and 6.

24. Open valve 1 slightly to slowly allow the glove box environment to re-enter the purifier. While doing this, turn valve 10 up to the glove box side, and open flow controller 12 to admit fresh Ar to the glove box. Monitor gauge 16 to insure that the glove box is never under-pressured.

25. When the purifier has been equalized with the glove box, and the glove box is not under-pressured, close flow controller 12 and valve 10.

26. Open completely valves 1 and 2 to restart the circulation of the glove box environment.

At this point, the gas purifier has been flushed of its built up Oxygen, however the air in the glove box is not any cleaner. It will take some time of circulating before the glove box's impurity concentrations will drop. Note that in this process, thermocouple 21 is closer to the heater than thermocouple 20, and so will always be hotter. It will therfore probably be the one to reach 250°C first. Also note that if thermocouple 20 does not come up and down in temperature during the flushing, then the oxygen remover was not completely oxidized.

3. Legend for the Diagram of the Glove Box System

This section serves as a legend for the attached diagram of the valves and connections in the glove box system. All of the numerically labelled components are explained below.

Label	Description
1–3	One-way valves, normally open.
4–8	One-way valves, normally closed.
9	Two-way valve that can be opened down to the pump, up to the glove box, or closed.
10	Two-way valve that can be opened to the large cylinder, to the glove box, or closed.
11	A metal T-connector in the pump's exhaust line.
12–13	Gas flow-controllers.
14–15	Pressure gauges that measure pressure in inches of Hg relative to atmospheric.
16	Pressure gauge that measures the pressure above atmospheric in the glove box, in inches of H_2O.
17–18	Pressure gauges that measure the pressure of gas remaining in the bottles in psi.
19	The two containers of oil that the glove box air can escape through.
20–21	The two thermocouples in the gas purifier.
22	The electrical connection to the heater for the Variac.