PROJET BOÎTE À GANTS

Document: User Guide
Prepared by: KHALID LAAZIRI

Contacts

- **KHALID LAAZIRI (PJAB 7467 – Room: 5094)**
- **JOËL BOUCHARD (Room: 4155)**

Introduction

A glove box is a sealed chamber (glass, acrylic...) maintained under dry atmosphere (Nitrogen, Argon...); it is designed to allow manipulating moisture and oxygen sensitive materials. In normal condition, a glove box should operate with Oxygen and Water concentration below 0.1 p.p.m.

This manual is based from the ***Glovebox_Operating_Instructions_ed11.2004*** mbraun manual

Glove box components

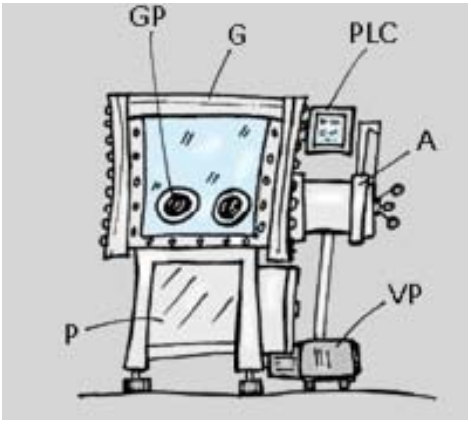


Figure 1

A standard glove box is composed of the following components:

G: "GLOVEBOX".

GP: "GLOVE PORT".

A: "ANTECHAMBER", small chamber to move things in and out the glove box.

VP:"VACUUM PUMP", the pump is used to evacuate the excess gas from the glove box, to reduce the pressure in the glove box and to clean the purification system after each regeneration.

P: "GAS PURIFICATION SYSTEM".

PLC:"PLC CONTROL", control surveillance system of the glove.

Gas purification system

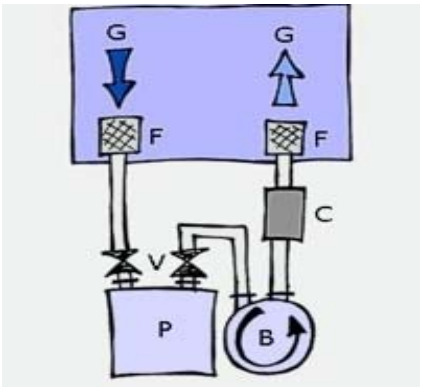


Figure 2.

The principle of a glove box consists on circulating dry gas through the chamber and a purification unit to remove the excess moisture and Oxygen.

G: "GAS FLOW", gas circulating between the glove box and the purification system.

F: "EXHAUST FILTER", dust filters connected at the inlet and outlet of the gas tubes.

V: "VALVES".

P: "PURIFIER UNIT", the purification is composed of two units,

- Oxygen purifier: Catalyst of polydispersive Copper particles.
- Moisture purifier: Absorption of water vapor by a "MOLECULAR SIEVE"

B: "BLOWER", 3-stage compressor who allows the re-injection of purified gas in the glove box.

C:"COOLING", allow the cool down the hot air expelled by the "BLOWER".

Purging the system

Displacing the ambient air from the system is calling purging. On principle, a system should be purged, when first in the beginning when commissioning the system; Or when the O_2 portion in the box atmosphere exceeds 100 ppm.

Caution:
A Glovebox system should be purged using working gas until the O2 portion within the box atmosphere has decreased to a value of < 100 ppm. Operating the system with higher oxygen value may result in damaging the gas purification system.

Figure 1: Example of Purge Gas consumption

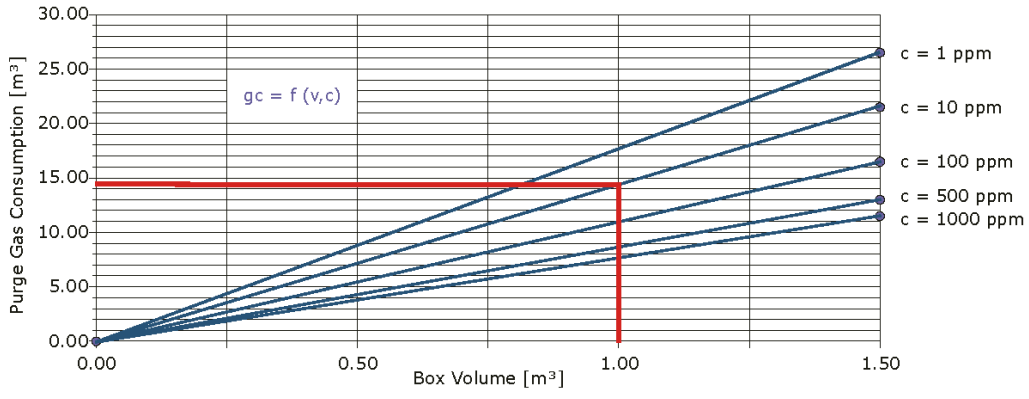


Figure 1 shows that if a purity of 10 ppm is required, then about 14.5 m^3 of purge gas is required for 1 m^3 box volume.

Pressure Control system

Definitions:

Box pressure: Current pressure prevailing within the glovebox.

Working pressure: Box pressure desired.

Working range: A fixed range within the working set points.

Working setpoints: Adjustable set points of the working range from -14.5 mbar to 14.5 mbar. When these set points are exceeded automatic pressure compensation is started.

***Manufacturer's settings: upper working setpoint +4 mbar; lower working setpoint -4 mbar*.**

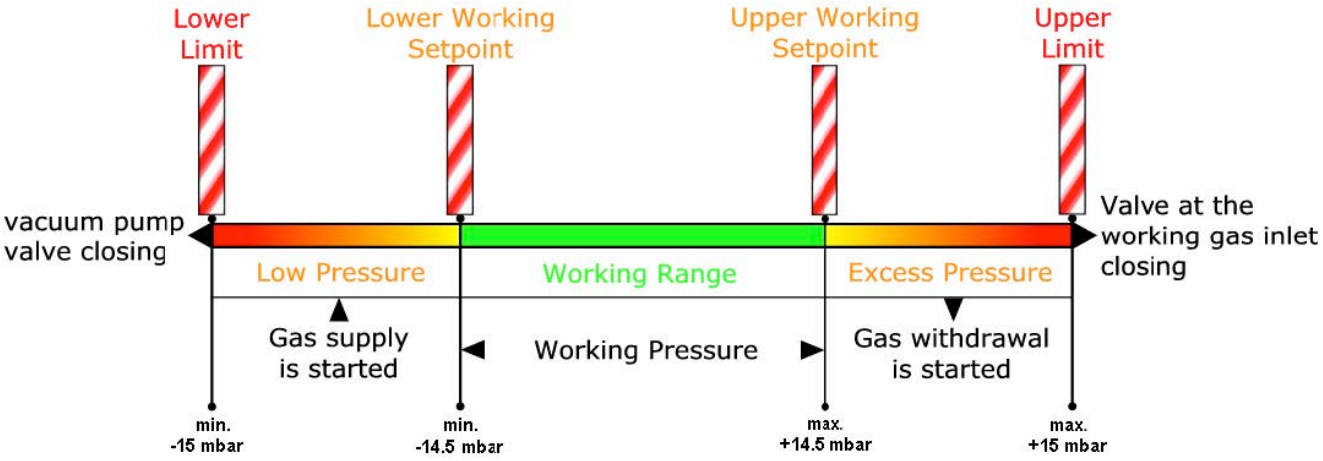
Limit setpoints: Adjustable maximum pressure set points outside working range for the system safety (-15 mbar to 15 mbar). When these set points are exceeded the gas limits, the valves are closed immediately.

The box pressure can be changed with a foot switch.

Pressing the right pedal increases the pressure within the working range.

Pressing the left pedal decreases the pressure within the working range.

The range of pressure system



Sensors

Oxygen sensor

Zirconium oxide Oxygen Sensors: This type of sensor is occasionally referred to as the “High temperature” electrochemical sensor and is based on the Nernst principle. Zirconium oxide sensors use a solid state electrolyte typically fabricated from zirconium oxide stabilized with yttrium oxide. The zirconium oxide probe is plated on opposing sides with platinum which serves as the sensor electrodes. For a zirconium oxide sensor to operate properly, it must be heated to approximately 650 degrees Centigrade. At this temperature, on a molecular basis, the zirconium lattice becomes porous, allowing the movement of oxygen ions from a higher concentration of oxygen to a lower one, based on the partial pressure of oxygen. To create this partial pressure differential, one electrode is usually exposed to air (20.9% Oxygen) while the other electrode is exposed to the sample gas. The movement of oxygen ions across the zirconium oxide produces a voltage between the two electrodes, the magnitude of which is based on the oxygen partial pressure differential created by the reference gas and sample gas. The zirconium oxide oxygen sensor exhibits excellent response time characteristics. Another virtue is that the same sensor can be used to measure 100% Oxygen, as well as parts per billion concentrations. Due to the high temperatures of operation, the life of the sensor can be shortened by on/off operation. The coefficients of expansions associated with the materials of construction are such that the constant heating and cooling often causes “sensor fatigue”. A major limitation of zirconium oxide oxygen sensors is their unsuitability for trace oxygen measurements when reducing gases (hydrocarbons of any species, hydrogen, and carbon monoxide) are present in the sample gas. At operating temperatures of 650 degrees Centigrade, the reducing gases will react with the oxygen, consuming it prior to measurement thus producing a lower than actual oxygen reading. The magnitude of the error is proportional to the concentration of reducing gas. Zirconium oxide oxygen sensors are the standard for in-situ combustion control applications.

Moisture sensor

Rules and usage policies

Before using the glove box, you must make a reservation via our website (**https://gcm-reservations-udem.phys.polymtl.ca:20443/**).

Strictly follow the rules and the glove box procedures.

Gloves

Gloves are important components of the glove box, so they should be treated with special care. Damaged gloves can lead to leaks in the glove box (therefore an increase in the oxygen and moisture levels). So everyone should follow the recommendations below

- Inspect the gloves you will be using for possible damage.
- Take off all jewelry and any watches.
- It strongly recommended putting white cotton gloves before putting your hands on the gloves (for sanitary reasons).
- It is recommended using Latex or Nitrile gloves inside the glove box when manipulating chemicals, in order to avoid cross-contamination and damage of the glove box gloves.
- Take careful precautions when manipulating sharp objects such as syringes, tweezers, and needles to avoid punching holes in the gloves.

Glove box workspace

The glove box workspace is an area that should be cleaned and emptied when your work is done. It is the area when you will do your preparations, prior to use it, you should follow this recommendations

- Before your working session. Please enter a plastic bag where to put your trash.
- Use an aluminum foil to cover the workspace. You should work on this cover area to avoid messing with the glove box.

- The workspace is not a storage area, so avoid leaving unlabeled product, glassware, and samples. Each user can leave inside the glove box a plastic box identified with a color tape corresponding to his group.
- Once your work is done follow the "Post-operation procedure"
    - Clean the workspace by removing the trash bag and the aluminum foil to outside the glove box.
    - Put back the chemicals to there place, put your stuff back in your basket and place it on shelf.

Transfer procedure

There is a strict protocol to follow in order to enter items such as glassware, lab ware, materials and so in the glove box. A part of regular lab ware items, you should talk to the supervisor prior introducing other type ot items.

The glove box is equipped with two types of antechambers, a Large and a small.

Small antechamber

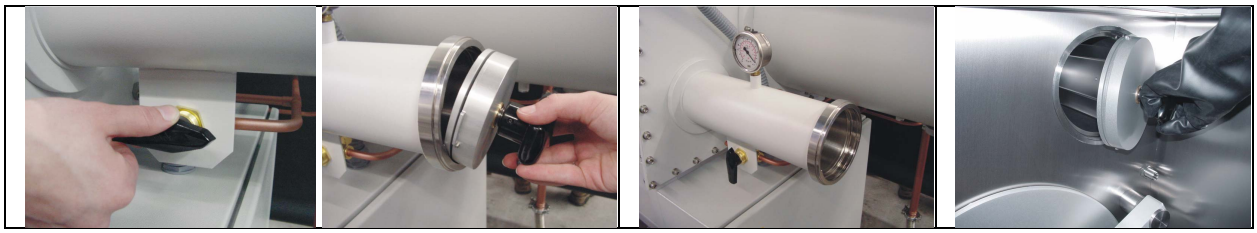


Figure 3 a, b, c, d

The small antechamber is operated manually. In order to transfer an item you should follow these steps:

1. Fill slowly antechamber with nitrogen by turning the control valve (Fig.3.a) to the "REFILL" position.
2. Turn the control valve to the "CLOSE" position. Open the antechamber door (Fig3.b) and place your item in the sliding trail, then close the antechamber door
3. Pump the antechamber by turning the control valve to the "EVACUATE" position.

4. Cycle between Evacuate (at lest 10 minutes) and Refill at least 3 times. These cycles allow removing excess moisture and Oxygen present when the antechamber was opened. Depending on the nature of the item to introduce into the glove box, there is a different level of precautions that are required (will be discussed further)
5. Put one hand in one of the gloves to open the indoor antechamber door, then take out your item, close the inside door of the antechamber (Fig.3.d). With your other hand turn the control valve to the "CLOSE" position.

The transfer procedure is drawn in this figure,

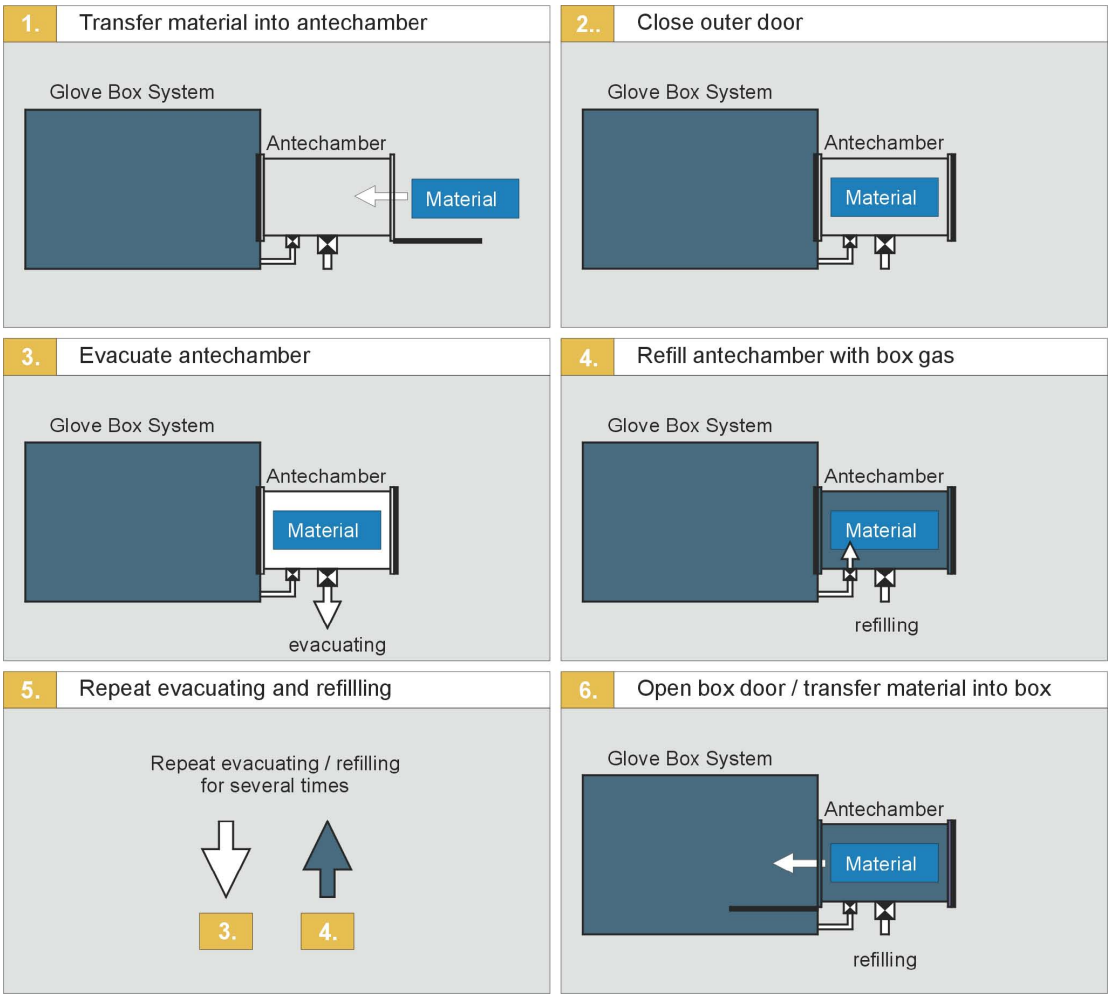


Figure 4

Large antechamber

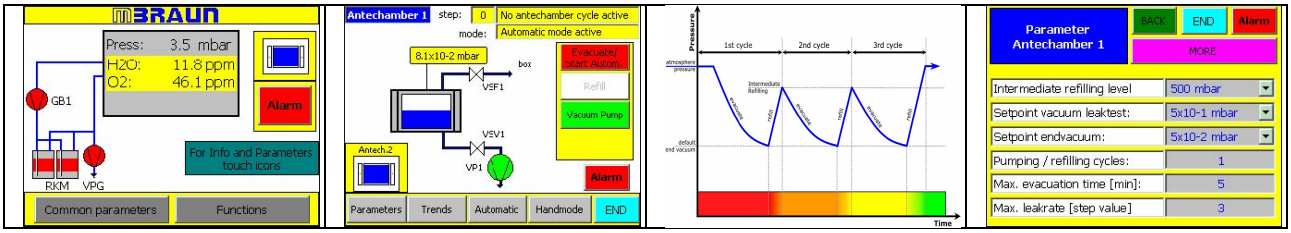


Figure 5. a, b, c, d

The large antechamber (main) is operated via the control panel "PLC", the transfer procedure is the same except that the Evacuate/refill cycle is done automatically by a predefined program on the PLC. It used to introduce bigger objects.

The transfer procedure is like this,

1. Push the blue icon at the right high corner in the PLC (Fig.5.a).
2. A new window will show the status of the antechamber, with a pressure reading in mbar. Empty box with a pressure reading of 10 mbar means that the antechamber is in vacuum. A filled blue color box with a pressure reading of 900 mbar indicates that the antechamber is filled with Nitrogen.
3. When the antechamber is filled with Nitrogen, you can open it to put your big item into the antechamber.
4. Close the antechamber door, and push the "**Evacuate/Start automatic**" button. The cycle will start automatically.
5. Wait until the cycle is over, then you can put your hand on the gloves and open the inside antechamber door to remove your object.
6. Close the antechamber door.

Transfer procedure for different materials

Liquids:

If packed under vacuum, or packed under a dry gas such as Nitrogen can be entered and opened into the glove box. For bottled chemicals packed under air, first loosen the bottle cap and remove the tape, then evacuate them in the antechamber, as soon as they are in the glove box tighten eh cap.

Equipment

In order to introduce ant type of equipment, micro-balance, heater... you should notify the glove box supervisor. Normally, if the equipment is clear to go into the glovebox, it should be pumped inside the antechamber for at least 24 hours.

Porous materials

Porous materials such as weight paper; KIMWIPES should be evacuated for extended periods of time.

Light materials

Light materials should be clipped during the transfer in the antechamber.

Glassware

Glassware should be introduced dry and clean. So you should clean your glassware and back them at 100C in an oven to eliminate water vapors, and then put them into the antechamber while they are still warm.

Closed glassware should never be introduced into the glove box.

Anhydrous solvents

Only anhydrous solvents are allowed into the glove box, and new bottles not used ones.

If your solvent is in a glassware type container, try to fill it to the maximum or complete the missing volume with nitrogen then close your container with paraffin or septum.

All volatile solvents such as alcohols, phenols are strictly forbidden to use in the glove box.