

School of Chemistry SOP For Operation Of Glove Boxes

School SOP for Operation of Glove Boxes:

The following SOP provides guidelines on how to adequately and safely operating a standard laboratory glove box and its associated devices.

General Information:

A glove box, also called a dry box, is a confined gas tight space allowing the manipulation of chemicals under an inert atmosphere. To some extent a glove box can be regarded as a substitute for conventional inert-gas Schlenk techniques employed on the laboratory bench. However, the best performance is obtained when combining glove box and Schlenk techniques. For this reason, every user should be familiar/comfortable with standard Schlenk techniques prior to use of a glove box.

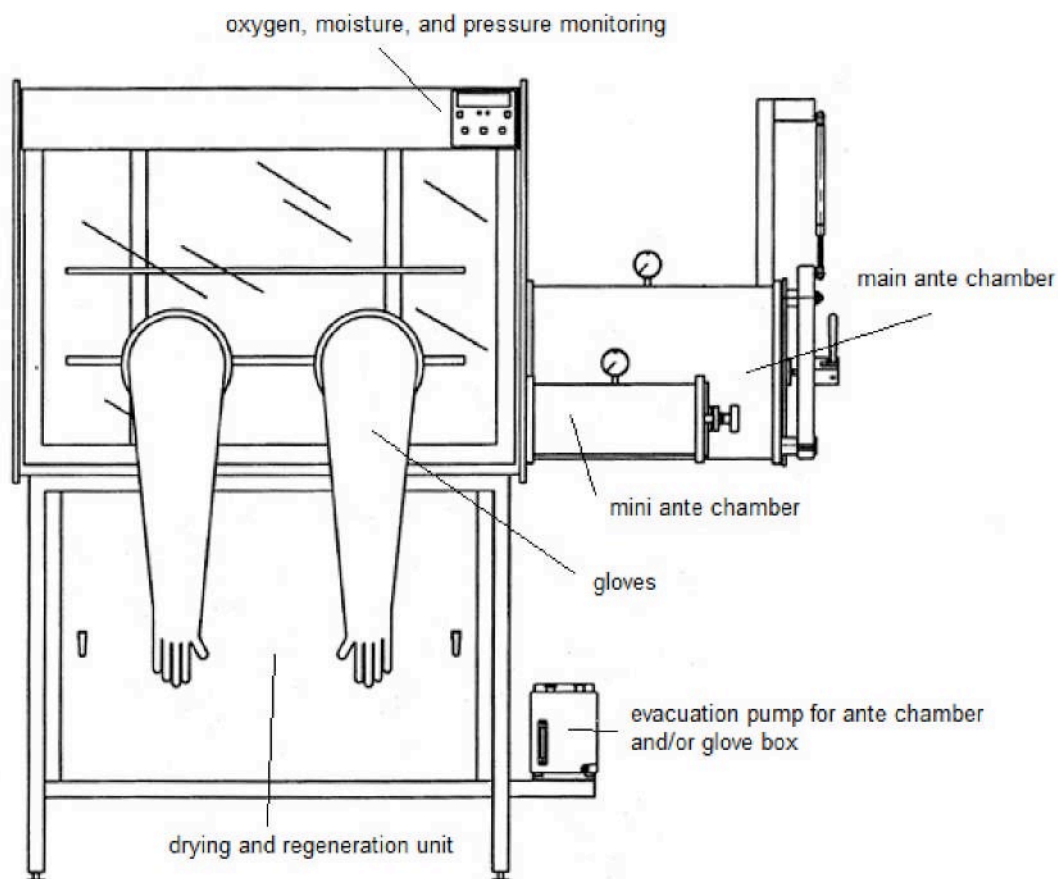
As described, a glove box is a confined volume containing an inert gas, different to air, allowing the handling of air and moisture sensitive compounds. In most cases, these boxes contain nitrogen or argon as an inert gas together with a purification unit to prevent the accumulation of moisture and oxygen within the box. The bigger the glove box, the more difficult it is to maintain a dry and air-free atmosphere. Even if the glove box is initially air-free and well setup, every user needs to take as much care as possible not to contaminate the glove box with air, moisture, and corrosive volatile chemicals. Therefore, every user needs to plan very carefully all manipulations to be performed in advance and to make sure that the desired compounds are absolutely air and moisture free before bringing them into the box. When working within such a glove box, great care must be taken in order to avoid any spillages and accumulation of waste – especially volatile and corrosive chemicals should in general not to be handled in a glove box as they damage the associated purification and generation unit.

In many cases, the glove box will appear to be the method of choice compared to conventional Schlenk techniques because less glassware and simpler reaction setups are involved, however, clean and safe glove box manipulation will turn out to be as time-consuming as the corresponding bench techniques.

School of Chemistry SOP For Operation Of Glove Boxes

General Overview:

The following figure shows the components of a standard glove box, similar to the ones installed in the A1 Laboratories at UCD.



Oxygen, moisture and pressure monitoring:

Every glove box is equipped with a water content measuring gauge and a pressure gauge. An operational glove box should contain less than 1 ppm of water. Some glove boxes also allow the continuous monitoring of the oxygen content, which should not exceed 1 ppm as well to guarantee an inert atmosphere. The pressure in a glove box is usually between 1 and 1.5 atm. Significant over pressure should be avoided by all means.

A glove box is designed to provide a gas-tight environment around standard pressure. To pressurize a glove box above 3 atm will cause the glove box to explode. Usually, the most pressure sensitive part is the gloves which easily rupture above atmospheric pressures and may cause sever harm to the operator.

Likewise, excess vacuum should be avoided. Therefore, the glove box should never be put under continuous vacuum. Most modern glove box devices do have an automated pressure control that balances the pressure increase caused by manipulation.

School of Chemistry SOP For Operation Of Glove Boxes

Evacuation Pump:

The glove box and the ante-chambers will be connected to a rotary vacuum pump. A single stage rotary pump can achieve pressures of about 1 torr, whereas a double stage pump can go down to about 10^{-4} torr. One of the most common reasons for bad vacuum with these types of pumps is too low an **oil level** – so the oil level needs to be checked regularly. In addition, the pump exhaust valve needs to be covered with oil in order to obtain a good seal. As a rough rule, any deviation in sound of the operating rotary pump indicates technical problems of which it should be taken care of immediately. For further specifications about rotary pumps, refer to the corresponding SOP.

Drying and Regeneration Unit:

The glove box is equipped with a recirculation system that exchanges the glove box atmosphere over an oxygen scavenger and a drying unit. These oxygen scavengers are based on copper (RIDOX and BTS catalysts) or manganese oxide, whereas the drying unit consists of molecular sieves (4Å or 5 Å). In order to maintain an inert atmosphere in the box, its volume is exchanged about 50 times per hour, which should allow for oxygen and moisture levels lower than 1 ppm respectively. When the level of O₂ and/or H₂O go beyond 1 ppm for an extended period of time, it is necessary to regenerate, see regeneration section.

Operating The Glove Box:

The following guidelines are a prerequisite to start working in a glove box.

- If the glove box is used by more than one person on a routine bases, a log book should be filled in advance to book the box for the desired time.
- If reactions are run over a longer time in the box, every user should keep in mind that the next person using the box will need enough space to perform its manipulations. Therefore, the working area in front of the gloves should be cleared and cleaned after every manipulation.
- Every item in the glove box should be placed and stored within a reachable distance. If necessary, tweezers can be employed to reach for distant objects.
- Every user should have properly labelled plastic containers inside the box where personal samples can be stored.
- After finishing work in the glove box, the gloves are wrapped inside-out and cleaned with a slightly watered tissue.
- After having performed work in the glove box, one has to make sure that the ante-chambers are left under vacuum.

Remember that some chemicals when stored in a glove box are inert, but when these are taken out of the box, they can react violently with the moisture and oxygen containing laboratory atmosphere (e.g. alkyl metals). Therefore, these items should not be directly exposed to the laboratory atmosphere. It has to be made sure that these highly reactive substances are not mixed with other chemicals and items within the glove box, and thus taken out accidentally.

School of Chemistry SOP For Operation Of Glove Boxes

Purging and Filling the Ante-Chamber:

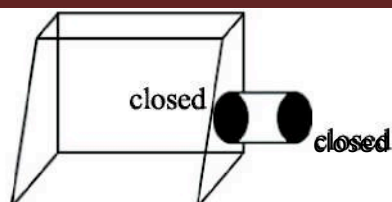
If one considers the ante-chamber a gas-tight system, the number of cycles of alternating vacuum application and inert-gas filling which are necessary to obtain a fraction of less than 1 ppm of air is given by the following equation:

$$R_{air} = P^n$$

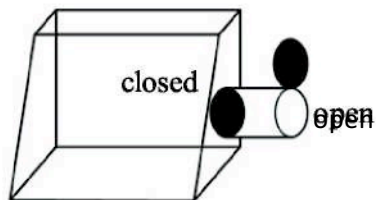
where R_{air} is the fraction of air remaining in the ante-chamber after n cycles using a vacuum pump able to achieve a pressure P . Therefore, for a typical ante-chamber of an approximate volume of 40 dm³, 3 cycles of applying moderate vacuum (1 torr) will result in a remaining air fraction of only 2 ppb.

- Check if the inner port door is closed, otherwise shut the inner door prior to evacuate the ante chamber (By default, the inner door has to be shut and the ante chambers set under vacuum).
- Shut off the vacuum.
- Start filling the ante-chamber with nitrogen until the ante chamber pressure gauge indicates approximately 1 bar, then open the outer door of the ante-chamber.
- Insert the required items in the ante chamber under a continuous flush of nitrogen. EVERY ITEM HAS TO WITHSTAND THE APPLIED VACUUM - **SOLIDS** are dried and brought into the ante-chamber in a closed vial. **AIR SENSITIVE SOLIDS** need to be put in a strong vacuum withstanding Schlenk flask. **NON-VOLATILE** and **VOLATILE LIQUIDS** need to be in a strong vacuum resistant flask (equipped with a teflon stopcock) containing 1 bar of inert gas.
- The outer door of the ante chamber is closed and the nitrogen is shut off.
- Evacuate the ante chamber for 15 or 5 minutes for the big or small ante chamber respectively. Flushing with nitrogen until 1 bar is reached.
THIS STEP IS REPEATED 3 TIMES
- After the above described cycles are performed, flush again with nitrogen until 1 bar is reached.

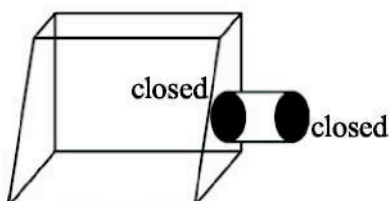
School of Chemistry SOP For Operation Of Glove Boxes



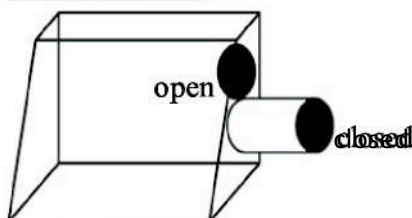
Ante chamber is under vacuum and then filed with nitrogen



If 1 atm of nitrogen is reached, the outer door is opened



Vacuum is applied, followed by flushing it with nitrogen – this step is repeated 3 times!



The inner door is opened and the items brought into the box – then the inner door is closed

Performing Manipulations in the Glove Box:

- Before you start working in the glove box, make sure that either all the steps listed under 4.2 are completed or if no items need to be taken into the box, make sure that the vacuum in the ante-chambers is shut off and that they are refilled with approximately 1 bar of nitrogen.
- Remove jewellery from your hands, as they can damage the gloves.
- Immerse your hands and arms into the gloves very slowly to avoid a sudden increase of pressure which will cause the glove box to automatically shut down. When you are able to handle the vacuum system manually (for example with an external foot pedal), apply a gentle vacuum in order to balance the pressure increase associated with the immersing the gloves into the box.
- Avoid manipulating sharp objects with the gloves, as the gloves may be damaged.
- After finishing working in the box, make sure that the ante chamber doors are closed – the ante-chamber is then put under vacuum. Switch the circulation system to standby (allows for lower nitrogen renewal frequency).

School of Chemistry SOP For Operation Of Glove Boxes

Taking Objects out of the Glove Box:

- Check if all the steps described previously are completed, otherwise check if the ante-chamber external doors are closed (if not, close them and perform 3 vacuum –nitrogen cycles as described previously).
- If the ante-chamber contains approximately 1 bar of nitrogen – make sure that the nitrogen supply is shut off.
- Once inside the glove box, open the inner door of the ante-chamber and place in there the items to be taken out from the box – make sure that they are properly sealed.
- Close the inner ante-chamber door – once, outside the box, apply a gentle flow of nitrogen when opening the outer ante chamber door.
- If all the items are removed, close the outer door of the ante-chamber and put the ante-chamber under vacuum.

Regeneration and Servicing:

Cleaning and Labelling:

The glove box needs to be cleaned after every manipulation. To clean the box inside, a box of Kleenex will do nicely. However, to avoid accumulation of moisture within the tissues, they should be dried in a vacuum desiccator prior to bring them into the box. In addition, the glass window should be cleaned at the outside on a regular basis, using a slightly watered tissue. As already mentioned, each flask and vial inside the box needs to be labelled properly. It is also suggested that every user has a plastic tray in the box where personal samples/items can be stored.

Regeneration:

To maintain an inert and dry atmosphere containing in maximum 1 ppm of oxygen and moisture respectively, the circulation unit needs to be generated at a level of about 0.95 ppm. Regeneration of the oxygen scavengers, namely BTS or RIDOX, is achieved by heating to about 200 – 300 °C under a stream of nitrogen, followed by flushing the scavenger with hydrogen (5 % hydrogen in 95 % nitrogen) to obtain again the reduced form. Molecular sieves can be regenerated by heating under vacuum. One has to be aware that the use of hydrocarbons and polar solvents presents a contamination problem of the regeneration unit. In particular, **volatile sulfides and halocarbons** should not be handled within the glove box if the regeneration unit isn't preceded by Linde 13X molecular sieves, which are able to trap these compounds. In order to use hydrogen gas as a regenerating agent, the user must be familiar with hydrogen gas SOP and the corresponding emergency procedures.

School of Chemistry SOP For Operation Of Glove Boxes

Diffusion of Air and Leaking:

The major part of air and impurity diffusion into the box is due to the gloves. It is estimated that about 59 ppm/h of atmospheric impurities diffuse through the gloves under stand-by conditions. When the glove box is in use, the diffusion of the impurities is increased by a factor of 10. Therefore, it is of crucial importance to choose the right type of gloves. In general, butyl rubber gloves are preferred over neoprene gloves as they show reduced diffusion of moisture and oxygen. To prevent impurity diffusion in stand-by modus, the glove ports should be sealed using glove port shuts.

If the values of oxygen and moisture within the glove box keep rising even after the drying unit and the oxygen scavenger had been regenerated, the box should be tested for leaks. Small leaks are detected by slightly pressurizing the box and cover the potential leak areas with soap solution – if bubbling is observed, the leak is detected.

Safety Guidelines:

- Gas cylinders for regeneration of the oxygen scavenger need to be fixed to the wall and freely accessible.
- The good functioning of the moisture and pressure gauges needs to be verified at least once a year.
- Be familiar with the MSDS of every substance you intend to work and/or to store in the glove box.
- When leaks are suspected to be present, increase pressure slightly to avoid contamination of the glove box – locate and fix the leaks immediately.

IF A SUDDEN PRESSURE BUILD UP IS OBSERVED, THE RECIRCULATION UNIT NEEDS TO BE SHUT
DOWN