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Lab Safety: Operating The Glove Box

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Overview

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A glovebox is an isolated enclosure that is designed to maintain an inert atmosphere. The manipulation of chemicals or apparatus is done via the use of gloves, which allow operation while still maintaining an inert atmosphere. A positive pressure is utilized to compensate for any passive leaks. Objects can be transferred in and out of the box using an antechamber that acts as an airlock between the box and the outside atmosphere[1]. A glovebox can have multiple pair(s) of gloves associated with it depending on the size of the box.

Principles

The glove box works by containing a closed circulation loop which utilizes a blower, plumbing, and a purifier to maintain H_2O and O_2 levels lower than 1 ppm. The atmosphere is purified by air circulation through a catalyst bed to remove moisture and oxygen. The catalysts include copper based particles to remove O_2 and molecular sieves to remove H_2O. After 3-6 months usage, the catalysts need to be regenerated by isolating the catalysts from the glovebox and exposing them under 5% H_2 in N_2 and heating to reduce the formed copper oxide to metallic copper. Certain volatile chemicals such as halogenated compounds and strongly coordinating species (thiols) should be excluded from the glovebox because they have a high propensity to poison the catalysts. Instead, experiments involving these types of chemicals should be conducted in a Schlenk line.

Initial training and proper operation are important for the safe use of the glovebox. All chemicals must be degassed and dried before being placed into the glovebox. Glass vessels or flasks must be evacuated before placement in the antechamber to avoid explosion due to the pressure difference. Sharps should be carefully handled and avoided if possible when working with the glovebox to avoid possible damage to the glove. Furthermore, secondary nitrile gloves must be worn to protect the glove when working with solvents. Aqueous solutions, alcohols, and acetone should not be used in the glovebox in order to ensure the glovebox environment is free of H_2O and O_2, which may help protect the glove over its lifetime. Compared to the glovebox, there are fewer requirements regarding the suitability of samples for the Schlenk line. However, there are other safety concerns when using schlenk line, such as ensuring glassware is not cracked, the possibility of over-pressurization of the glass schlenk line, and the use of liquid nitrogen in the pump trap could lead to the possible condensation of oxygen, if a leak exists in the schlenk line.

Procedure

1. Transferring Objects into the Glovebox

1. Make sure both doors are closed (this should be the default position). If the chamber is under vacuum, fill it with nitrogen by turning the valve to fill.
2. Open the outer door, and transfer the object in the chamber. Slide the object as deep as possible, which makes it easier to reach it from inside the glovebox. Close the door isolating the antechamber from the ambient environment. DO NOT over tighten the door, go only a quarter-turn further.
3. Once the doors are closed, turn the valve to evacuate the chamber, and wait until the pressure is at an acceptable vacuum level. Then fill the chamber with nitrogen, by turning the valve to refill. Repeat this cycle at least 3 times.
4. Once the purging and evacuation cycles are complete, fill the chamber with nitrogen, and then open the inner door to the glovebox. Remove the object from the antechamber and place in a safe location in the glove box. Promptly close the door to the antechamber.
5. With the doors closed, turn the valve on the antechamber to vacuum, and leave it there. This is the default position of the antechamber when not in use.

2. Transferring Objects Out of the Glovebox

1. Make sure the antechamber is filled with nitrogen. If you are unsure about the state of the chamber, evacuate and refill the chamber with nitrogen at least 3 times.
2. Open the inner door, and transfer the object into the chamber. If the object includes chemicals, make sure it is properly sealed. Then close the inner door to the antechamber.
3. Open the outer door and remove the object(s).
4. Close the outer door and turn the valve to evacuate. The default condition for the antechamber is to be under vacuum.

3. Chemicals and Apparatus

1. Make sure all of the apparatuses that you transfer into the glovebox are dry.
2. Any chemical that is taken in the glove box must be dry and deoxygenated. It is best to use DriSolv solvents. If these are not available, it would be preferred to use solvent bottles which have not been opened outside of the glovebox.

3. Take only the required amount of chemicals into the glovebox, full reagent bottles MUST NOT be taken in (unless the reagent has to be permanently stored in the box).
4. Use 20-mL vials to transfer solids and non-volatile liquids. Wrap the top of the vials with lab wipes and use a rubber band to hold the wipe in place. This allows the air above the solid/liquid to be evacuated.
5. While transferring volatile liquids, use Teflon stoppered flasks.
6. Be extremely careful while using needles and blades in the glove box. The gloves are extremely delicate, and any puncture can compromise the atmosphere in the box.
7. Once you have finished your work, take out all the apparatuses promptly and clean the area.
8. Store any chemicals that have been synthesized in an appropriate location within the box and label it appropriately.
9. Let the person in charge of the box know if any of the glovebox-dedicated consumables are running low.

4. Solvents

1. While taking in solvents, make sure to use the Teflon-stoppered round-bottom flasks, and only use dry deoxygenated solvents from the solvent stills or as ordered from chemical suppliers.
2. All the solvent bottles stored in the box should be capped all times.
3. Solvent vapors can damage the gloves, and hence any solvent spills must be promptly wiped with lab wipes, and then removed since volatile solvent vapors will remain.
4. Place the solvent bottles back in the appropriate location once you have finished using them.

5. Waste Disposal

1. Dispose of the waste in properly labeled waste beakers.
2. Recap the needles after use before disposing of them.
3. If the waste container is full after you use it, take it out of the box, and dispose of it properly in the lab waste, and promptly replace the waste container in the glovebox.
4. Clean up any spills on the glove box floor and dispose waste in the misc. waste.
5. The liquid waste bottle MUST be capped at all times.

6. General Tips

1. Do not insert/remove your hands from the gloves quickly. Doing so quickly will cause a sudden pressure change in the box, which can lead to ambient air from the external environment accessing the glovebox through any micro pores on the gloves.
2. Be extremely careful while using needles and blades in the glove box. The gloves are extremely delicate, and any puncture can compromise the atmosphere in the box.
3. Make sure the chambers are always left under vacuum after use.
4. Shut off the blower using the main screen when handling amines, phosphines, or any other volatiles, since they can damage the catalysts.
5. Keep a logbook of all glovebox activity including maintenance performed on the glovebox.
6. Conduct maintenance (vacuum pump oil change, regeneration of purification system) on a regular schedule.

Applications and Summary

Gloveboxes are essentially devices in the research laboratory for the manipulation of air- and water-sensitive chemicals that are not easily worked with on a Schlenk line. Gloveboxes are particularly useful for the transfer of chemicals into vessels that are incompatible with Schlenk line assemblies. The proper operation, good housekeeping, and maintenance of a laboratory glovebox ensure that sensitive experiments can be conducted without exposure to oxygen and water.

References

1. Ashby and R. D. Schwartz *J. Chem. Educ.*, 1974, *51* (1), p 65
2. Glovebox Use Guidelines, University of Houston, 2009.
3. Standard Operating Procedures, Bartlett Group, University of Michigan, 2016.