

Standard Operating Procedures for the Use of Dryboxes

A drybox is a highly specialized equipment and there are at least three different manufacturers who make dryboxes. In addition, several users have custom designed and installed various other components as accessories. It is difficult to write a general SOP for their use. The following general guidelines have evolved from years of experience in the use of dryboxes. They should be strictly followed, especially if community use is envisaged.

- **Have one person in charge of the drybox.** This person should know how to operate all components of the box, know what are the best practices in the use of a drybox, and should be familiar with the regeneration of the oxygen and water scavenger systems. This person should also know where and how to contact the manufacturer in case help is needed. This person may seek the help of a second member of the group, who will assist in cleaning, maintenance and regeneration of the box. The second person also must develop the required expertise and should be able to take over the responsibilities if the person in charge is absent or when that person leaves.
- **All new users should get thorough training before independently operating the box.** First few times it is best that someone knowledgeable (preferably the person in charge) watch over your shoulders. There is very little room for error, and at most caution should be exercised when operating the box.
- **Everyone who is using the box should read the instructions that came with the box thoroughly.** In addition, read all accessory manuals that are used. All prudent practices in dealing with high pressure/vacuum equipment must be followed in the use of cylinders and pumps that are attached to the box. There are several shutoff valves used for control of gas and vacuum in side the box. Be sure that you know what exactly happens when you either open or shut one of these valves. If you don't, do not touch that valve! This is especially important during the regeneration process since there is a risk of pressure build up or decompression during this process. Even though the regeneration process can be run automatically we recommend that during the entire process someone is present, just in case, one of the automatic pneumatic valves fail. If this happens the box could be compromised and recovery could be very expensive.
- **The level of oxygen and water should be checked periodically if no sensors are installed, and the system should be regenerated at the first indication of deterioration of the inert atmosphere.**
- **Pay particular attention to the quality and integrity of the gloves.** We recommend the thicker variety (available from all manufacturers), even though some dexterity is lost in their use.
- **Use the best quality nitrogen or argon.**

- **Extreme care should be exercised not to contaminate the inside of the box.** Nothing should be spilled. In case of spillage clean up immediately and remove the waste from the box as soon as possible. Spilling reactive chemicals (especially volatile or sublimable ones) should be avoided at all cost. Handle solvents and reagents with great care.

- **Minimize the use of volatile solvents.** Keep all vessels closed. Syringes and disposable pipettes used should be deposited in a waste jar that can be tightly shut (so that evaporation of solvent from these can be minimized) and should be removed from the box as soon as possible. Halogens, sulfur and phosphorus containing materials are particularly harmful to the regeneration system.

- **Drybox is not a storage place.** Keep only essential items needed for current use.

- **If you suspect that the box has been compromised, let everyone, especially your supervisor, know.**

Specific Instructions

- Some specific instructions for a double drybox in room 3069 Evans (used primarily for organic and organometallic synthesis) follow. The safety committee recommend that each group develop its own specific set of instructions for other special applications.

- **Drybox should be under positive pressure at all times.** The small antechamber should always be left under nitrogen, by evacuating it at least for one minute per each cycle three times. Whenever the big antechamber is left under air, it should be kept under reduced pressure (-10 to -20 in Hg) to avoid accidentally opening the inside door by mistake. If the status of the antechamber is not sure, it should always assume it is under air.

- **Wear cotton gloves whenever you work in drybox.** This will prevent moisture from your hands from permeating the gloves, and protect the drybox gloves from your nails.

- **You should not touch the floor of drybox with gloves.** When you need to pick up something small from the floor, you should use long tweezers. Some crashed glasses like broken pipettes on the floor will puncture the gloves. It is very hard to find the holes on the gloves.

- **Materials from the freezer should be warmed up to room temperature before taking in through the antechamber and opening inside the box.**

- **The caps of all the bottles in drybox should be closed tightly and sealed with electrical tape to avoid contamination of inert atmosphere by solvent vapors.**

- **Everything you pump in should be dry.** Some material or equipment that is likely to degas over a long period of time or to release large amount of water (e.g., Celite, kimwipe, paper, silica gel, TLC plates, cork, glass-wool or other porous materials) should be pumped down overnight after drying process in a vacuum oven as described below:

Pretreatment before pumping in overnight

Molecular sieves and Celite for drybox use should always be kept at about 150 °C in a vacuum oven. After use, refill them for next pumping in.

Kimwipe should be dried at about 70 °C in a vacuum oven for a few days. Otherwise, leave in under vacuum in the antechamber over the weekend.

Silica gel and TLC plates can be dried at about 150 °C in a vacuum oven overnight.

- **Syringe use in drybox**

When you use needles or cannulas in drybox, you must be extremely careful not to make holes on the gloves. Pay attention to the tips of needles all the time. While you do not use them, they must be inserted into rubber stoppers. If possible, try to use an automatic microliter pipette instead a syringe. Since its battery lasts only a short time, it should be turned off immediately after use.

Waste disposal

Remove your contaminated waste (chemicals, solvent, syringe, etc.) from drybox as soon as possible using a plastic bag in drybox. Clean glass waste can be disposed in a waste container inside the drybox. Empty this as needed.

You must keep the drybox clean.

Do not leave anything (e.g., used pipettes, contaminated spatulas, vials containing chemicals, reagents, solvents, rubber bulbs, etc.) except a reaction in progress on/around the stirring/hot plate for next person.

Clean your working area on/around the stirring/hot plate and the balance, by wiping off solid waste and solvent residues.

Try to hold your personal stuff in boxes with your initial. Do not put too many chemicals/supplies on a center rotary wheel, so nothing falls down during the rotation.