

# Standard Operating Procedure: Glovebox Operation

Last Updated 11/08/16 by Selene Li

Two gloveboxes are located in Rm. 429. The big double box (NEXUS) is operating under in-house nitrogen, and the small box (MO-10-M) is operating under argon by associating a gas cylinder. The user operations of these two gloveboxes are slightly different as described below in detail.

**The gloveboxes should only be used (initially) with the aid of experienced operators.**

## **Pre-Treatment of Equipment and Chemicals:**

Equipment and chemicals need to be dried before transporting into a glovebox. Glassware and metal spatula can be baked in the ovens overnight, or glassware can be flame-dried before pumping into the glovebox. They can also be pumped under vacuum overnight in one of the antechambers before transporting into the glovebox. Other experimental supplies, which may be destroyed by heat, must be pumped under vacuum overnight in one of the antechambers.

**Solid chemicals** have to either be dried under vacuum in a desiccator over  $P_2O_5$  overnight or vacuum-dried with heat on schlenk lines for sufficient amount of time before pumping into the box. The caps on the containers need to be detached, and the mouth will be rubberband-wrapped with kimwipe while pumping. If they are purchased as anhydrate and non-volatile solids packed under inert gas, they can be pumped into the box in the original intact package.

**Hydrate or volatile solids** need to undergo 20-quick-refill-vacuum cycles, and they should not be pumped in an antechamber with normal procedure or left overnight to prevent water or chemical from destroying the Edwards direct drive pump.

**Liquid chemicals** should be collected from a dry or wet distillation using a thick-wall high-vacuum solvent bomb, unless they are purchased as extra dry reagents packed under inert gas in sureseal bottles. High-boiling liquid samples need to be dried by at least 5 cycles of freeze-pump-thaw, and low-boiling liquid should undergo at least 20 cycles of gas exchange to make sure minimal amount of  $O_2$  remained before transporting into the box. Liquid chemicals CANNOT stay in any of the antechambers overnight, and they need to be pumped in by the standard 3-cycle procedure described below.

See the glovebox point-person with more questions.

## **Big Double Box Operation:**

To transfer materials to the glovebox using either the large or the mini antechamber, you should refill the chamber with the gas associated with the box and evacuate it with the materials inside the chamber for 15 to 20 minutes. The refill and evacuation routines of the large antechamber for the big box are controlled by the system computer with the control panel; these routines are operated by the manual control valves for the mini antechamber. Before proceeding any of these routine, make sure that both inside and outside antechamber doors are closed. (See an experienced operator or the user manual if you have more questions.) This refilling and evacuation should be performed three times total before the materials accessing the interior of the box.

Before opening either chamber, make sure that the antechamber has been properly cycled and is closed to the inside and outside so as to not expose the interior to the outside atmosphere.

# Standard Operating Procedure: Glovebox Operation

Only dry chemicals, flame-dried or oven-baked glassware and properly degassed and dried solvents should be taken into the glovebox. When the antechambers are not under use, both chambers should be under evacuation.

Before exposing any solvent or chemical inside the glovebox, the circulation blower should be turned off, and the purifier (catalyst) should be closed. After the exposure of solvents or chemicals inside the glovebox, the atmosphere within the box should be purged to displace the lab's existing atmosphere using the purge routine. The duration of the purge is a judgement call, please see an experienced operator if questions rise. The purifier should be open immediately after the purge routine to keep the box atmosphere dry and air-free. The normal range of oxygen level is 0.1ppm, and the dew point ideally should be below -50.

## **Small Box Operation:**

Same principles mentioned above are applied for the operation of the small box to keep the box atmosphere dry and air-free.

If the antechambers are not under use, they should be under vacuumed. Three 15-to-20-minute cycles should be performed before any dry materials accessing the interior of the box. The difference is that both refill and evacuation routines are controlled manually by the valves on the control panel for the large and the mini antechamber of this small glovebox. Before performing these routines, make sure the pressure sensor is connected to the corresponding gauge monitoring the chamber.

An argon gas cylinder is associated with the small box. The pressure of this gas cylinder should be checked regularly to insure a proper gas volume is present. If the pressure is below 500 psi, the replacement of the cylinder needs to be done immediately. For the replacement of the gas cylinder, please obtain the aid of an experienced user, and do not attempt it on your own until you have been aided through the process at least once. After the replacement, the box needs to be purged for 20 minutes before turning on the circulation blower and opening the catalyst.

## **Personal Protective Equipment:**

Safety glasses, nitrile gloves, and clean lab coats designated for the use of gloveboxes are required for the use of the [big](#) glovebox.

Safety glasses, nitrile gloves, and long sleeves are required when operating with the [small](#) glovebox.

## **Waste Disposal:**

After use, the working area in the box should be wiped clean with appropriate choices of solvents, and any trashes generated during the process should be pumped out of the box immediately after. Any hazardous waste should be disposed properly according to the standard operating procedure.

## **Material Safety Data Sheets:**

Call the ORCBS (517-355-0153) or see lab copy for samples in questions if available.