Standard Operating Procedure

Task: Bringing Things into the Glovebox
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Background:
Antechambers are the primary way to exchange materials in ambient atmosphere with those under an inert gas. Proper use of antechambers will minimize atmospheric gasses entering the glovebox, maintaining the inert environment crucial to air and moisture sensitive reactions.

Training Requirements:
- Laboratory safety training
- Glovebox training with Brandie

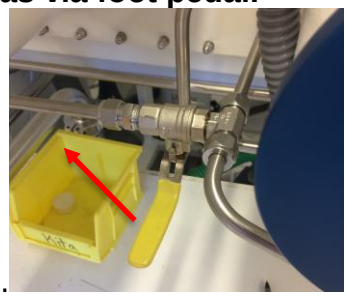
Potential Hazards
- Exposure of pyrophorics to air
- Possible inhalation hazard from volatiles through the pump
- Chemical Hazards
- Burns from hot glassware
- Equipment damage (see below)

Materials Needed:
- Log sheet
- Vary by materials being brought in (See **Guidelines)**

Procedure:
- *Small antechamber*
    - Fill out log sheet and flip tag.
    - Check and ensure that the large antechamber vacuum and nitrogen valves are closed.
    - Refill the small antechamber with nitrogen then turn the valve to the “closed” position (straight up and down).
    - Open the outer antechamber door and insert object to be brought in (see guidelines below) then close the door.
    - Turn the valve to vacuum.
    - Once full vacuum is reached, evacuate chamber for at least **30 seconds.**
    - Refill the antechamber to **–5 in. Hg** (-0.2 bar) then immediately return the valve to vacuum and evacuate the chamber as before.
    - Complete 5 evacuate/refill cycles.
    - Upon completion of the fifth cycle, refill the antechamber completely, turn the valve to the “closed” position, open the inner door, bring objects in, close the door, and return the chamber to **vacuum.**

- *Large antechamber*
  - Fill out log sheet and change tag on chamber. ***Note that the large antechamber refills from the "wet" box. If bringing items to the "dry" box please ensure that the atmosphere in the "wet" box is free of volatiles that could contaminate the dry box. You should purge the "wet" box if you are uncertain about the "wetness" of the atmosphere.***
  - Check and ensure that the small antechamber vacuum/nitrogen valve is closed.

  - Refill the large antechamber with nitrogen by opening the yellow valve. **Note: Open slowly to prevent significant drop in box pressure and compensate pressure drop with extra gas via foot pedal.**



  - Open the outer door and insert object to be brought in then close the door.
  - Open the black vacuum valve. **Note: Open slowly to avoid pressure differential "wind" scattering chemicals and supplies.**

  - Once full vacuum is reached, evacuate chamber for at least **20 minutes.**
  - **Once large chamber is pumped down, place the small antechamber under active vacuum.**
  - Close small chamber vacuum/nitrogen valve and refill the large antechamber to **-0.15 bars** then immediately return the valve to vacuum and evacuate the chamber as before.
  - Complete 3 evacuate/refill cycles.

- Upon completion of the third cycle, refill the antechamber completely, turn the valve to the “closed” position, open the inner door, bring objects in, close the door, and return the chamber to vacuum.

- *Filling out the log sheet:*

			Small Chamber			Large Chamber							
User	**Date**	**O_2 Level (ppm)**	**1**	**In?**	**Out?**	**1**	**2**	**3**	**In?**	**Out?**	**Purge Start**	**Purge Stop**	**Comments**
BL	5/3/16	1.7				8:40	9:15	9:35	✔	10:00			Pipettes
BL	5/3/16	2.0			3:20						3:00	3:10	CH_2Cl_2
AC	5/3/16	<0.1	11:30	✔	11:45								pentanes

  - Small Chamber**:**
    - 1: Time first cycle was begun, follow small chamber pumping procedure.
    - Self-check: "In": Has the material been pulled into the glovebox? Did you return the antechamber to active vacuum?
    - Self-check: "Out": Time the user pulled an object out through the small chamber, exposing the antechamber to atmosphere.
  - Large Chamber:
    - 1: Time first cycle was begun, follow large chamber pumping procedure.
    - 2: Time second cycle was begun.
    - 3: Time third cycle was begun.
    - Self-check: In: Has the material been pulled into the glovebox? Did you return the antechamber to active vacuum?
    - Self-check: Out: Time the user opened the large antechamber to atmosphere to remove an object from the glovebox.

Guidelines for bringing supplies into the glovebox

- *Glassware, stirbars, and metal*
  - These objects should be dried before being brought into the box
    - Most of these items can be dried in the 120º C oven for 4 hours or flame dried then brought in (e.g. bombs, Schlenk flasks, vials, pipettes, stirbars, spatulas). **Caution: Burn hazard.**
- *Plastic/rubber items*
  - **Note: These items can retain water!**
    - Plastic and rubber should be warmed in the warming oven before being brought in if possible (e.g. NMR caps).
    - Pump in overnight at least and for 24-48 hours if possible.
- *Paper items/glass microfiber filters*
  - **Note: These items retain large amounts of water!**
    - Kimwipes: Remove interior plastic, then warm carefully in 60º C oven for 48 hours then pump on in large antechamber for 24-48 hours.
    - Glass microfiber filters: Warm in 120º C oven for 24 hours then pump in overnight.
- *NMR samples*
  - NMR samples prepared outside of the glovebox that have a headspace of air should not be brought into the glovebox! Transfer sample to a vial and follow protocol below.

- Sealed NMR samples prepared in the box can be brought back into the box by parafilming the cap before pumping in. NMR tubes other than J-Young should not be brought back in the box. **Note: Do not leave samples in antechamber for long periods of time as NMR caps and parafilm are permeable.**

- *Chemicals and solvents*
  - Note: All chemicals should be labeled with the date they were opened, the date they were brought into the box, and the initials of the person bringing them in.
    - *Solids*
      - Solids should be dry before being brought into the box.
      - Solids must not be prone to sublimation (e.g. ferrocene, Co_2CO_8)
        - Solids prone to sublimation can be degassed in a Schlenk flask on the line while in a dry ice/acetone bath and then brought into the box.
      - Vials or bottles containing solids from other boxes should be tightly sealed with electrical tape before being transferred.
      - Vials or bottles containing dry, air exposed solids should be covered with a Kimwipe secured with a rubber band before being pumped in.
      - Reactions run outside of the box can be pumped down to solids on the line before being brought into the box (be sure to clean off any grease with hexanes before pumping glassware into the box). *Limit time spent in the box with objects that have not been thoroughly dried.*
        - *Schlenk flasks*: pump down fully on line and bring evacuated flask into box
        - *Bombs*: reactions taken out of the box run in dry, degassed solvents in a Teflon-stoppered flask under an atmosphere of nitrogen or argon can be pumped directly back into the box without evacuating.
          - If the reaction is under a headspace of air, or wet, oxygenated solvents were used, the reaction must be pumped down and evacuated before being brought into the box.
        - *Vials*: pump down in a vac-shack (aka airfree vacuum chamber) and bring entire evacuated apparatus into the box.
  - *Celite, alumina, sieves* ***(See Drying Celite, alumina, sieves SOP).***
  - *Liquids*
    - Liquids should be degassed and dried according to standard procedures before being brought into the box in a **Teflon-stoppered flask** (no ground glass stoppers) **(See Degassing SOP)**.
      - *Oily Materials: Those materials synthesized outside of the glovebox should be pumped down with high-vacuum and placed in the antechamber with a Kimwipe*
      - *Bulk solvents*: should be brought into the box directly from the solvent system in a Straus flask.
      - *Deuterated solvents*: should be degassed in a bomb by repeated freeze, pump, thaw cycles before the evacuated flask is pumped in.

- *Sure-Seal bottles*: seal the cap with electrical tape, pump the bottle in, turn the purifier off, take the cap off, purge for 20 minutes, and turn the purifier back on. Newly purchased solvents that are prepacked and capped under nitrogen can be pumped in directly after receiving once they are secured with electrical tape.

- *Important:* A capped vial should never be brought into the box unless it is capped under nitrogen

Risks to Equipment:

Common Hazards	Consequences	How to Avoid
O_2	Box exposed to large amounts of Oxygen and atmospheric water	• NEVER open both antechamber doors at once • Properly cycle antechamber and keep track of antechamber usage with the box log sheet/tags • Do not to open antechamber unless you are absolutely certain that it has not recently been exposed to air
Sublimation	Coat antechamber with chemical and damage vacuum pump	• Follow protocol for bringing in solids prone to sublimation
Pressure differential "wind" caused by evacuating/refilling antechamber quickly	Knock over and spill chemicals or break glassware (e.g. NMR tubes, cuvettes)	• Evacuate and refill antechamber slowly when it contains items likely to be blown around
Antechamber doors not fully closed	*Outer door*: Damage vacuum pump *Inner door*: Box under negative pressure; gloves sucked into box and possible O_2 exposure *Both Doors*: O_2 exposure	• Ensure that door handles are secure and nothing is protruding from the antechamber
Refilling large antechamber too quickly	Box under negative pressure; possible O_2 exposure	• Refill the large antechamber slowly and use the foot pedal to increase box pressure if necessary
Leaving antechamber under atmospheric pressure	Possible O_2 exposure	• Keep antechamber under vacuum when not in use
Pumping down "trash"	Damage vacuum pump	• Do not leave anything in the antechamber that is not coming into the box • Keep track of antechamber usage with the box log sheet/tags and communicate with labmates

Related SOPs:

- Degassing
- Pyrophoric chemicals
- Waste
- Setting up Vacuum Traps
- Liquid O_2
- Drying Alumina, Celite, and Sieves