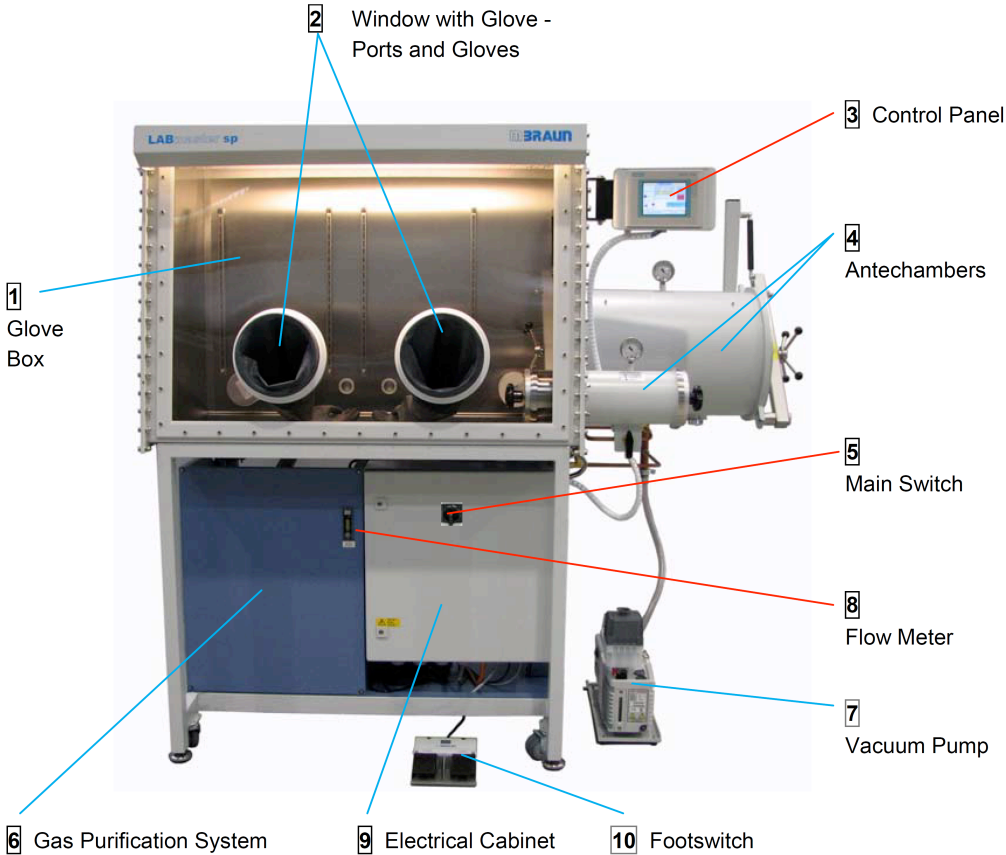
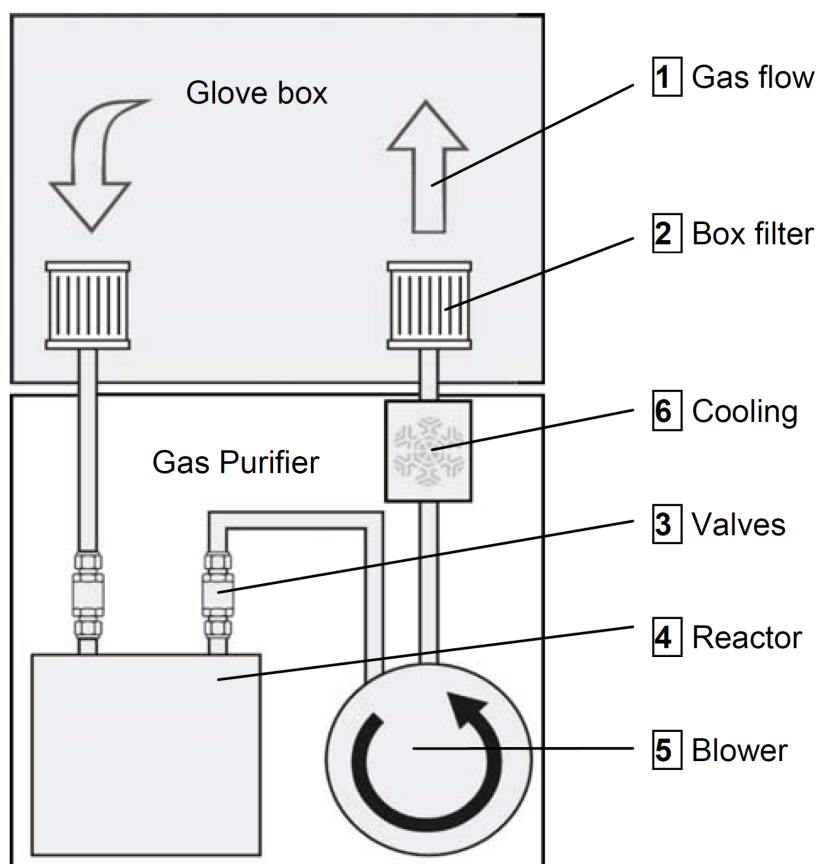


Glovebox Policies and Procedures (updated October 2016)

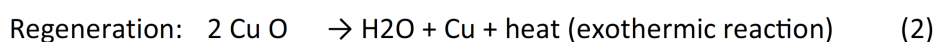
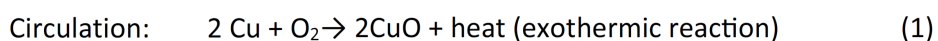


Glove Box with Gas purification platform Labmaster SP/DP:

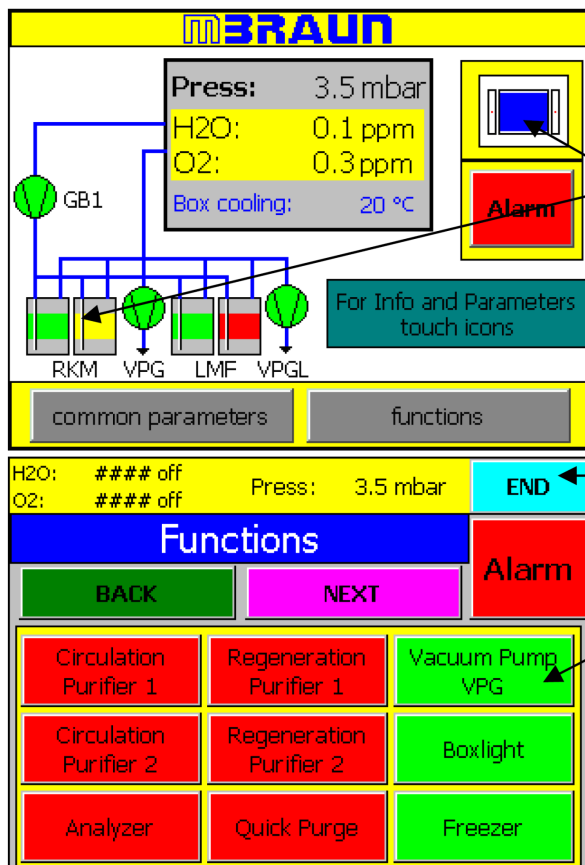
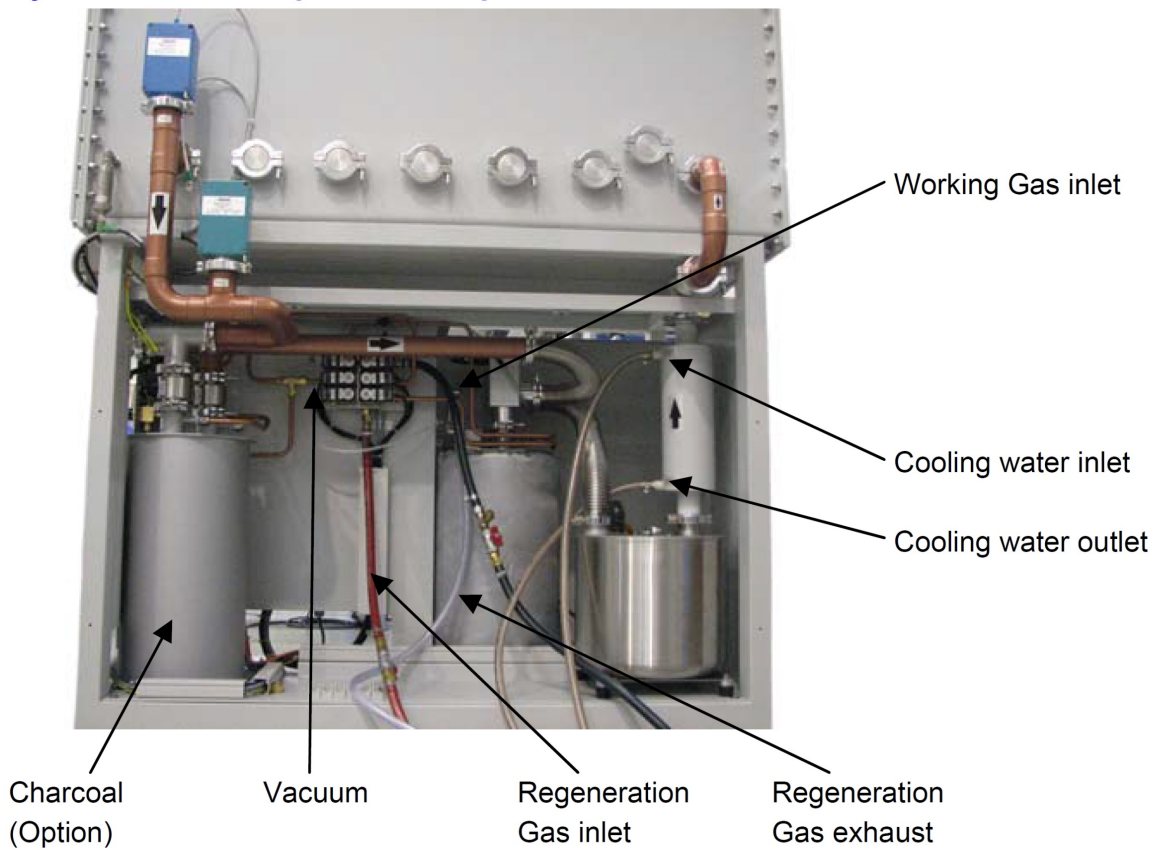




The used copper catalyst is a finely dispersed activated/reduced copper oxide deposit on porous aluminium support. It consists of approximately 30% copper on the carrier material. During circulation the inert gas flows through the reactor column where copper reacts with the oxygen to copper oxide according to equation (1). During regeneration, the copper oxide is reduced to copper by means of hydrogen according to equation (2).



Systems with Gas purification platform Labmaster SP/DP:



Icon Buttons:
The Icon Buttons are a pictorial representation of the item that it controls

Navigation Button

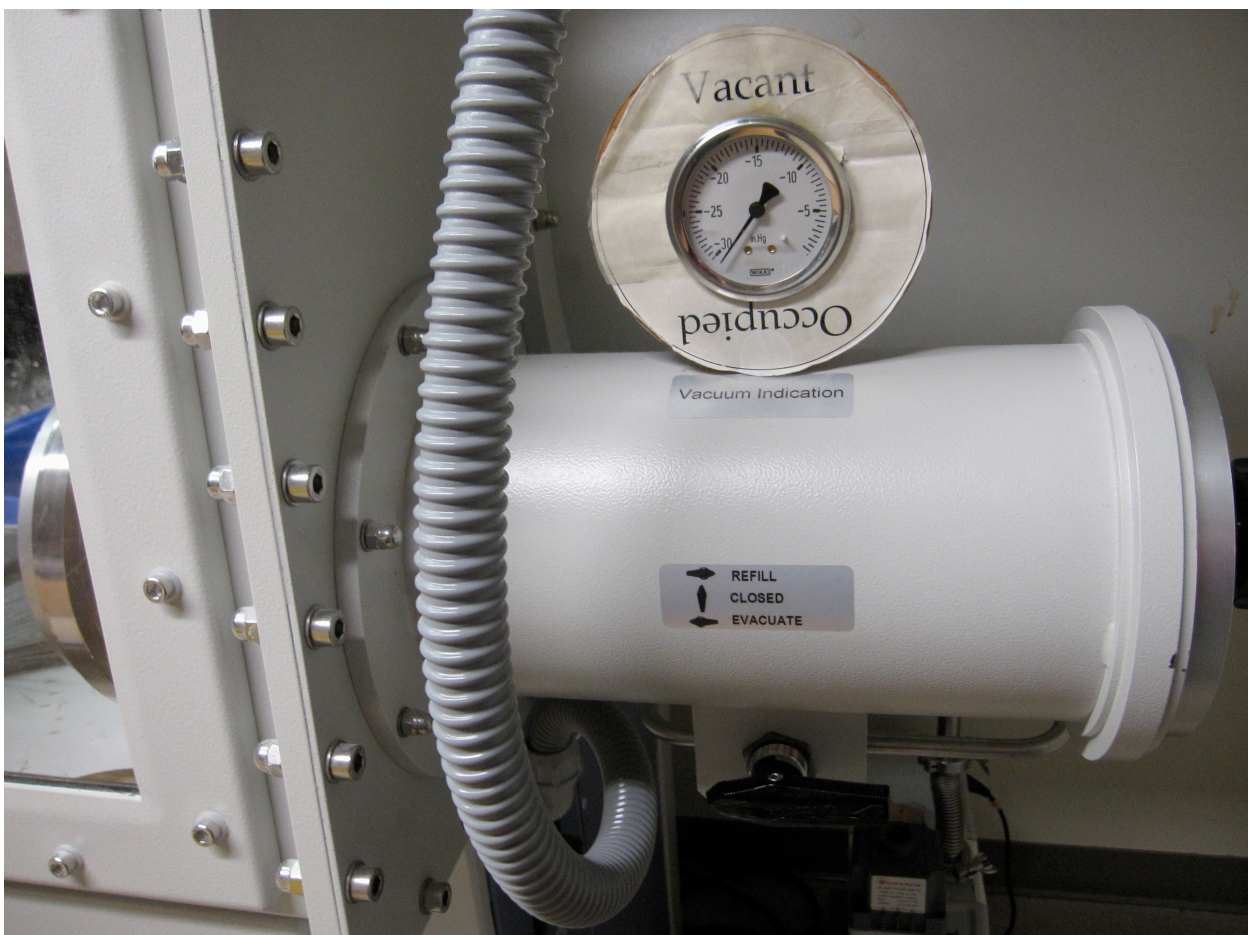
Function Buttons:
The Function Buttons are labelled with an appropriate description for its function.

1 All new coworkers must go through a training session.

RULE NO 1: IF THERE IS A PROBLEM, REPORT IT IMMEDIATELY!

SAFETY: BE AWARE THAT THE GLOVEBOX AND THE PUMP VENT INTO THE ROOM AND VENTILATION IS NOT AS EFFICIENT AS A FUME HOOD. IN OTHER WORDS: ALL FUMES INSIDE THE BOX WILL EVENTUALLY END UP IN THE SURROUNDING ROOM (PURGING WILL EXCHANGE THE ENTIRE GLOVEBOX ATMOSPHERE). IT IS ADVISABLE TO LEAVE THE ROOM WHILE PURGING TOXIC FUMES BUT PLEASE STAY NEARBY (E.G. WATCH THROUGH THE DOOR WINDOW).

2 A typical pump down procedure



- a. Start a new entry in the log book.
- b. Fill the antechamber with inert gas.
- c. Set the valve to the closed (up) position and open the outer door.

- d. Place your equipment inside the antechamber and close the outer door.
- e. Evacuate the antechamber and switch the sign to “occupied” while in use.
- f. After at least 5 minutes refill the antechamber with inert gas, repeat evacuation/refill cycle at least twice (three times 5 minutes), then open the inner door.
- g. Bring your equipment into the glovebox and close the inner door.
- h. When you are done working in the glovebox, open the inner door and place your equipment and trash in the antechamber.
- i. Close the inner door and set the valve to the closed position.
- j. Open the outer door, remove your equipment, and close the outer door.
- k. Set the valve to evacuate and sign out in the log book.

3 Working inside the glovebox

- a. When working in the glovebox use of nitrile inner gloves (size XL) is suggested in order to protect the glovebox gloves, especially when working with corrosive/”sticky” compounds..
- b. Make sure you always maintain a positive pressure.



4 Box Solvents and Reagents

- a. All solvents should be dried and thoroughly degassed.
- b. Receiver bottles must be oven dried when placed into the antechamber. Only

Strauss flasks and specific bottles with a Teflon lined cap are allowed for solvents..

- c. Don't store large amounts of solvents, especially volatile ones like dichloromethane (!) in the box.
- d. Try to avoid the use of protic reagents such as alcohols, phenols, and amines, as well as halocarbons, phosphines, arsines, SO₂, and SO₃. Most of these compounds damage the catalyst irreversibly.
- e. No mercury inside the glovebox, since if it is spilled it will amalgamate the metal on the bottom of the glovebox.

Note: other reagents in addition to those mentioned can be damaging to the catalyst. In general, keep all solvent and reagent vessels tightly closed (ideally wrap the cap with electrical or Teflon tape), and try to minimize the amount of time they are open.

5 Useful Box Materials

- a. The people in charge of the glovebox (primary responsible person and also the backup & support person) will try to maintain a common supply of vials, pipettes, gloves, weighing boats, Kim wipes (baked out), grease etc..
- b. If you find it necessary to add any of these materials yourself, please watch for the following points.
- c. Any kind of glassware, also items such as spatulas, should be heated in the oven in the glovebox room for a minimum of 1 hour, preferably overnight.
- d. If for whatever reason you need to bring into the glovebox glassware that wasn't heated, it is recommended to evacuate it overnight before glovebox entry.
- e. Weighing boats can enter the box by a normal pump down procedure and do not require drying.
- f. Dry paper in the oven overnight and also evacuate it overnight before glovebox entry.

6 Precautionary Items

- a. The upper pressure limit on the control panel must be maintained at 8-10 mbar.
- b. Whenever working with any of the reagents that can possibly contaminate the atmosphere or damage the catalyst (see above) or volatile solvents TURN OFF the circulation, then purge the glovebox atmosphere (about 100 psi) when you are done, and turn the circulation back on. Even if the solvent/reagent is not damaging the catalyst its vapors will saturate the solvent trap.
- c. Prior to purging the glovebox atmosphere make sure that there is a sufficient amount of operating gas (Ar).

- d. Left glovebox only: Turn circulation off if the glovebox box port pump has to be turned off.
- e. Only the people who are responsible for the glovebox may regenerate the purifier units (with a mixture of 5 to 10% H₂ and Ar as the carrier gas).
- f. Everyone who is using the glovebox should be trained on how to reset the glovebox freezers.

7 Housecleaning and Storage of Chemicals

- a. Storage containers for each researcher's chemicals will be provided by the person in charge of the box.
- b. All containers and vessels containing chemicals must be clearly labeled and placed in their appropriate place (i.e. no random storage of materials on the floor of the glove box). Samples on the glovebox floor will be disposed off.
- c. Take out your waste on your own. Be careful with pyrophoric waste (e. g. paper tissues used for cleanup of pyrophorics can ignite easily).
- d. If you use something, put it back. If you exhaust something, replace it or notify the person in charge of replacing it.

8 Miscellaneous

- a. Turn off the hot plate after you are done.
- b. If you damage the glovebox gloves let the people who are responsible for the glovebox know IMMEDIATELY.
- c. Clean the balance if you make a mess.
- d. Do not write on the window of the glovebox or the ground; use paper or something else.
- e. Do not move the balance.
- f. Return personal chemicals or glovebox equipment into its proper location.
- g. It is also recommended that users check the Ar tank before and after working in the glovebox.
- h. Use of protective cotton or nitrile gloves is required to protect the gloves of the box (long finger nails (!)).
- i. Please make sure to take any items off your hands / arms (e.g., watches, Aggie rings, etc.) that could possibly damage the glovebox gloves - prior to start working in the box.
- j. Be extremely cautious when working with silica (or similar fine powders). It will not only cover the inside of the box with hard to remove dust but is also very bad for the pump. The best way to bring such materials into the box is to introduce them into the antechamber in a tightly sealed flask, which is evacuated (fill the flask in your hood, evacuate it, and then take it to the glovebox).