

## Glove box

Glove box can be very useful if you know how to. They need careful maintenance. Always follow the rules. If you are not sure, ask **Xile!**

The glove box is meant to provide the convenience for working under inert atmosphere, i.e., without O<sub>2</sub> and H<sub>2</sub>O. But before it can do it, you need to make sure you do not bring even trace of O<sub>2</sub> and H<sub>2</sub>O into the box. The general rule is that everything is bad for the glovebox. With a few exceptions, nothing goes into the glove boxes as it is.

Solvent scales:

I. Solvents **compatible** with the purification catalysts: Pentane, Hexane, Benzene, Toluene, Heptane, and other hydrocarbons.

II. Solvents **bad** to the catalysts: THF, Ether, DME, etc.

III. Solvents **very bad** to the catalysts: Methylene Chloride, Acetonitrile.

IV. Solvents **extremely bad** to the catalysts: Pyridine, Methanol, Chloroform, DMSO, DMF, Pyridine.

V. **Never** use as solvents: acids, water etc.

1. The box can work under three modes: circulation (blower on), purging, or neither. Under circulation, the box atmosphere goes through the catalyst for purification. The box should be under the circulation mode when not in use and when no solvents are used.

Type I solvents can in principle be used with the blower on if they are to be used for a prolonged period (for example when using toluene for a cooling bath during a reaction of several hours).

But it is still best to turn off the blower when simply adding or transferring them, since their vapors may otherwise permeate the glove box and catalyst and show up in your other experiments (or those of your colleagues).

Always switch the circulation off when you are working with solvents/vapors that are toxic to the catalysts, i.e., solvent II to IV. After working with the blower off, purge the box to regenerate the atmosphere. Normally a timed 30-min purge is required. If you just used small amounts of THF and ether, then a 20 min purge is enough. Do not turn back to circulation without purging.

2. Regenerate the box every 3-6 months (depending on the performance) to reactivate the catalyst. Regenerate the activated carbon. Change the pump oil every three months.

3. Do not leave anything open in the box for too long. Solution reactions running in vials should be capped.

4. If the box atmosphere contains other vapors, purge the box for 15 min before running a routine NMR, and 30 min before making samples for a very clean NMR.

5. Be careful of solvent contamination. If you are using ether, do not open a bottle of other solvents because you don't want those solvents to contain ether as well. Purge generously if you are concerned.

6. For big antechambers, evacuate/refill for 3 times, every time 15 min. For small antechambers, evacuate/refill for 3 times, every time 5 min. Exception can be made in special circumstance (e.g. transferring crystals), but use good judgements.

7. All glasswares to be used in the box have to be heated at 120 degrees Celsius overnight before entering the antechamber. Ideally they should have been in the oven for 6 hours or more.

8. Vials to be used in the box need to be heated at 120 degree overnight before entering the antechamber. The same is for glass pipettes.

9. Only chemicals packed under N<sub>2</sub> or Ar can enter the box with the caps on. For other chemicals, the cap needs to be loosely screwed on (half a turn) so that gasses can pass, but it won't fly off.

10. All liquids to be used in the box have to be degassed and dried. A 3-cycle freeze-pump-thaw procedure is recommended for degassing. The drying method depends on the specific compound. Transfer such liquids to the box using air-free glasswares with tight seals (e.g. Teflon O-ring joint). If you are using Schlenk glasswares, you normally need to evacuate the vessel and seal the glass joints with electric tapes. This is because when pumping the antechamber, the seal can break off if the inside pressure of the vessel is not zero.

11. Reactive reagents such as Organo Li, Zn, Mg compounds normally come with a sure-seal bottle. You could take them in as it is. When you work with them, make sure the waste is treated properly because they will cause fire in the open air immediately (e.g. the second you open the antechamber). Alert another group member when you do this, and proceed with caution, so that you are prepared and helped in case of fire.

12. Sensitive compounds purchased from commercial sources, such as Ni(Cod)<sub>2</sub>, often come in sealed ampules, so you could take them in as it is. Open the ampul inside the glove box (take care not to damage the gloves with broken glass!) and transfer the contents to a vial that can be sealed air tight. Accidents happen once a while so even in the box you need to be careful how you store the compounds.

13. Label everything! Make sure the label last. For pyrolytic and explosive compounds, make sure you have multiple labels.

**14. Report anything abnormal with the box. If something is not working, do not leave it.**

15. Solvents from the box come from the solvent purification system.. Here is the general procedure:

16. Heat a Straus flask at 120 degrees Celcius overnight. These flasks are available in 3220 and typically you can find one already in the oven. If you use that, be sure to ask the person who put it there on that day and be sure to replace it. Connect the flask to the system and evacuate it. Wait until it is cool, and follow the solvent purification system procedure to get the solvents. Take in the flask (which should be sealed). Store the rest solvent over activated molecular sieves. *If you are suspicious about the water content of the solvent, test it with Karl Fischer titration.* If it is indeed too wet, notify the person responsible for the solvent system, as it is possible that the

columns require replacement. It is also possible the sieves used for the storage have saturated and require replacement.

17. Use regular white caps for routine use; use polymer lined caps when you need good seals and when you are heating with solvents inside the vial.

18. Always make sure to take out all of the trash you generate while working in the glove box. **Be aware that small amounts of highly sensitive compounds left on the trash (e.g. spatulas or syringes) may react with air as soon as you remove it from the glove box.**

19. Thiols and Phosphines are bad for the catalyst. When working with them, make sure you turn the blower off. After the work, purge for at least 15 minutes before turning the blower on again.

20. Do not put things containing wood in the box. They often contain a lot of water that is hard to remove.

### **BOX RULES: general**

21. **If you make a mess, clean it up.** There is absolutely NO REASON for a spill to go uncleaned. This goes for a spilled reaction, or spilled alumina. If you need to clean it up with solvent and are weary of using the tweezers to push kimwipes around (as to not dissolve the gloves), use disposable gloves worn over the gloves of the glove box. If you need to clean the glass, use acetonitrile then purge the box as usual.

22. If you use solvents, replace them. When the bottles have maybe a couple cm left above the sieves, bring more in. At the very least, start collecting the solvents.

23. Use tape to additionally seal vials that contain liquids.

24. Always keep some NMR tubes in the oven, and when we are running low on tubes, make sure to clean yours and put them in the oven.

25. If someone is using the large antechamber and you want to use the small antechambre, close off the large as you open the small to the outside and as you backfill with nitrogen. This will make things go faster with bringing stuff in, so that no extra cycles need to be added.

26. When two people are in the box, make sure to manually correct the pressure using the foot pedal. Often the change in pressure turns off the vacuum and the circulator. Check and turn it back on.

27. Try and keep the vacuum ports clean. At the very least, the immediate area. Bumping happens and they will never all be clean, but we should be able to trust that if our stuff bumps, we did not contaminate our sample.

28. Try and notify your boxmates ahead of time if you plan to do some crazy chemistry that requires a lot of purging or nasty solvents- for example, so that they know to plan their experiments around this. Your solvents might contaminate their chemistry!

29. If you get ANY solvent pumped up in the vacuum for the antichambers, or the trap went dry overnight and there is non-frozen solvent, the pump oil needs to immediately be changed.

30. If you have any doubts or questions about whether you are doing something that is potentially harmful for the box, ask your colleagues or Xile (for example what's the best way to bring in mercury?)