

Working correctly with the nitrogen line

Working with the nitrogen line belongs to the daily routine at LCSO. A few simple principles are essential to do it correctly.

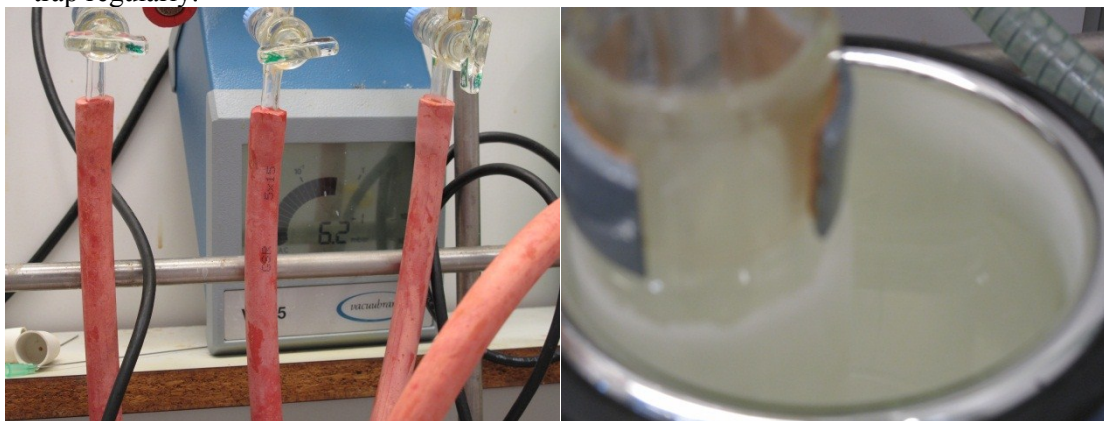


1) Installing the line.

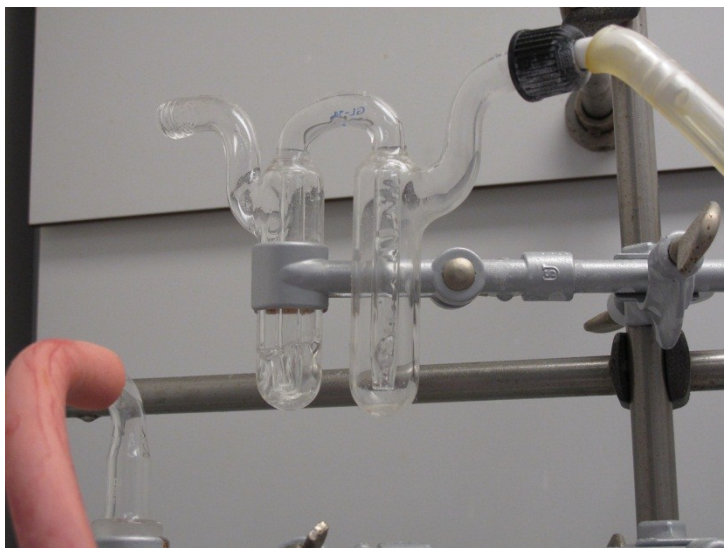
- Always check that your line is clean, change regularly tubing for vacuum and nitrogen

The two essential media of the line are vacuum and nitrogen:

- **Vacuum:** A good vacuum is essential for a clean work with the line. A tight line should always have a vacuum lower than 1.0^{-1} mbar. If this is not the case, you probably need to tight up your vacuum tubing and re-grease your joints. You may also need to change the oil of the pump: this should be done at least once a year.
- **Vacuum trap:** A liquid nitrogen trap is essential to get a good vacuum. Always check that the nitrogen level is high enough. If you break down the vacuum, **always remove the liquid nitrogen**, else explosive nitrogen could condense in the trap and lead to accident! Empty your trap regularly.



- **Nitrogen** (or Argon): Argon is more expensive, but has the advantage to be heavier than air, allowing easier manipulations. Always use dry inert gas. The gas is dried by passing through a drying agent, like drierite. Remember to reactivate the drying agent in the oven once a year. The bubbler at the end of the line allows you to control the gas flow. **When filling up a flask under vacuum, the oil in the second compartment should never go back completely to the first compartment, else air will enter the line.**



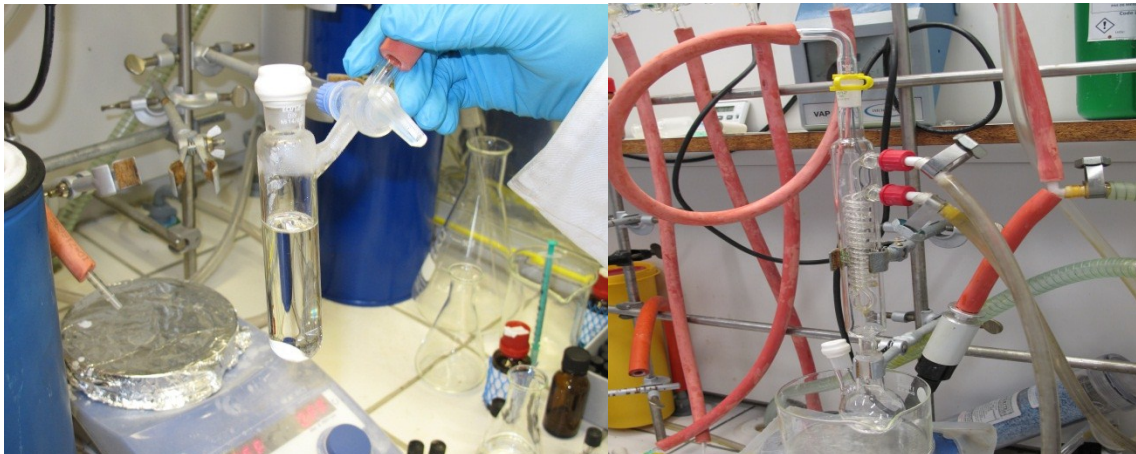
2) fundamental techniques: Septa and Schlenk

There are two ways to work with the line:

- The **septum technique**: it can be used only for not sensitive reactions. The only connection to the line is via a syringe needle. The needle is connected to the tubing via a cut syringe. The transfer of all chemicals takes place via syringe (see section on syringe technique). This technique is practical, but does not prevent completely air and moisture to enter the reaction flask.

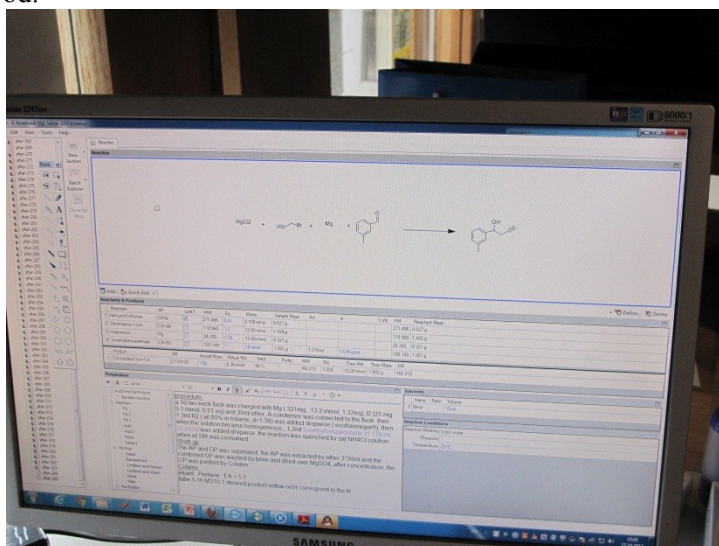


- The Schlenk technique: In this case, the flask is always connected directly to the line via tubing, and not via a needle. The reaction flask needs to have at least two necks to allow the transfer of chemicals, and it can be either a dedicated Schlenk flask or another multi-neck flask. Glass joints need to be secured by Teflon or grease (put grease only on the upper two-third of the joint to avoid contaminating your reaction). Only new Septa can be used in the evacuating phase, as used septa will let air enter the flask! The Schlenk technique is much better for sensitive reaction, as it assures a tight system with always a sufficient overpressure of inert gas.



3) Planning

You should start your reaction only when you know exactly what you will do. Your entry in the lab journal should be ready, and you can summarize the most important information on a piece of paper that you can take to the hood.

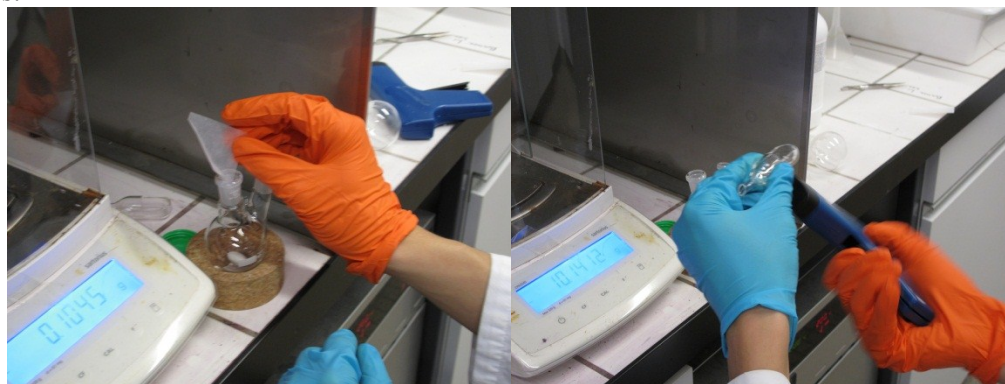


4) Transfer of chemicals to the line

- Use only dry glassware on the line! The glassware needs to be dried in the oven over night. It can be also kept for a few minutes in a dry box. For very sensitive reactions, the flask needs to be flame-dried under vacuum directly on the line.



- **Transfer of solid:** If the reagent is not too sensitive, it can be weighted directly on or next to the balance. If it is medium sensitive, it can be transferred directly to the reaction flask by opening the side-neck under nitrogen overpressure. If it is very sensitive, he needs to be transferred in the glove box. The used flask needs to have a switchable vacuum joint, as for example in a Schlenk, to allow an air-free transfer to the Schlenk line. Use an electrostatic gun to help transferring the solid. Go then fast to the line and evacuate/refill three times with nitrogen. Finally, clean all what you used (weighing flasks, spatula, balance) and if toxic transfer the residues to the special wastes.





- **Transfer of liquid:** Always transfer liquid under an overpressure of inert gas (see syringe technique section). Once finished, seal the septum with a teflon tape, close the flask and seal it with parafilm. If the reagent is in a flask without septum, flush it with inert gas before closing it.



- **Solvent from solvent system:** Take the solvent from the solvent system in a dry flask. Follows the rule of use exactly. Before connecting the flask to the line, flush the tube with inert gas, then add the needle. Always check that the needle is not blocked, then put it in the septum. Use then standard syringe or canula technique to transfer the solvent.



5) Taking note of events

During all the process of the reaction, take note of events, so that you can use it later to write a good journal entry.

