

Glovebox TR1.14 Guidelines

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How to use the antichambers

- Don't introduce in the antichamber closed **containers** with oxygen: insert them **opened or filled with inert gas**.
- If you introduce closed containers, consider that the cap could pop out because of the vacuum and spread your compound in the antichamber, take proper countermeasures.
- **Never introduce solvents containing water.**

Detailed procedure for inserting small objects:

1. Make vacuum in the antichamber (this pulls the internal door in the proper position);
2. refill with nitrogen;
3. **leave the tap in closed position** (if you leave it in refill position acts like a big hole in the GB);
4. open the external door and introduce your objects, all the containers should be **opened**. *E.g.* Petri dishes should be inserted opened;
5. close and make vacuum, wait until the vacuum gauge goes under its minimum and some time more;
6. refill **slowly** (the gas flow could throw your stuff around);
7. when the gauge approaches the max pressure switch again to vacuum (don't leave it at max pressure unless it's the last cycle);
8. repeat two more times (a total of **three vacuum-nitrogen cycles** for the small antichamber);
9. refill, open the internal door, remove your object, close the internal door and **leave the antichamber under vacuum** (doors are leaky).

Detailed procedure for extracting small objects:

1. **Make vacuum** in the antichamber (even if you think it's under nitrogen, it is not: doors of the small antichamber are leaky);
2. refill with nitrogen, open the internal door, insert your stuff, close the internal door taking care it goes in the right position;
3. make vacuum in the antichamber (this pulls the internal door in the proper position);
4. refill with nitrogen and **leave the tap in closed position** (if you leave it in refill position acts like a big hole in the GB);
5. open the external door and remove your objects;
6. close the door and **leave under vacuum**.

Procedure for inserting big objects:

- For using the big antichamber follow the same instructions as above but **six vacuum-nitrogen cycles** are suggested.
- If possible, heat the big objects and introduce them while they're still hot.
- Leave the big antichamber in **static vacuum**.

Before starting working

- **Record the date, your name and H₂O, O₂ concentrations** and all significant events (regenerations, malfunctions...) on the glovebox log book.
- If you're going to use the spin coater or **solvents**, even if in small amounts, which damage the oxygen removal catalyst (methylene chloride, acetonitrile, alcohols, amines), **stop the circulation** purifier while working then, when you finish, make a **quick purge** (10-40 min, in the Functions menu of the GB controller) and switch on again the circulation purifier (nitrogen is much cheaper than a replacement for a damaged catalyst).
- If you are using solvents whose vapours accumulation can damage your products, additionally keep the quick purge functioning for all the duration of your experiment.
- When starting using the GB **lower the internal pressure** to approx 3-5 mbar (using the GB with too high pressure (14 mbar) makes the circulation purifier and the analyser to shutdown).
- For using the **spin coater vacuum pump**: first switch on the small pump and then open the vacuum tap on the back of the GB (remember to close and switch off later).

- If you notice that the oil level of the small pump is low, refill it. We're in charge of the small pump maintenance.
- Use **lab coat and clean gloves** (the GB gloves are not clean, at all), if you have a watch, bracelet or rings better to remove them (for reducing the risk of making holes).
- Always use aluminium foil for keeping clean the internal part of the spin coater.
- If you're going to work with solvents put **gloves on also in the internal side** of the GB (do it, they're not uncomfortable, indeed they tighten the black gloves on your fingers giving a better grip).
- If the big or small antichamber pressure is not very low, report it to *Chromatography, Thermal Analysis & Electrochemistry Unit*: rubber rings need replacement.

After using the glovebox

- **Label everything** and keep your products closed and **in a box with your name** on it, periodic cleaning will trash things happily & arbitrarily, keep clean and you won't have to complain in vain.
- **Throw the wastes** in a container and take it with you when you finish working, leave as little mess as possible for the next user, otherwise your wet wastes will go on contaminating the glovebox and poisoning the oxygen removal catalyst.
- Leave the spin coater lid open, this way the droplets and residual solvent vapors can get removed by the purge process.
- Leave the **small antichamber under static vacuum**.
- Leave the big antichamber in static vacuum.
- If you used the spin coater remember to first **close the vacuum tap** and then to **switch off the small pump**.
- When finished using the GB **increase the internal pressure** to approx 10-12 mbar.

General guidelines

- Remember that the small antichamber doors have leaks, vacuum-nitrogen cycle the antichamber even if it shouldn't be needed: **When in doubt - pump it out!** Do not make any assumptions!
- Keep your samples open only when necessary. This is to avoid GB contamination and contamination of your samples.

- When weighing materials in the glovebox, static electricity is a big problem. To minimize this disturbance use the antistatic gun and steel weighing boats.
- Never keep needles around, throw them in the proper container, needles and cutting edges (*e.g.* glass sharp edges, scissors) are gloves' worst enemies.
- Don't use the antichamber vacuum for removing solvents, the pump has no solvents trap!
- Enter and exit your arms in the glovebox **slowly** so that the overpressure doesn't get negative or too high (over 14 mbar, causing the GB circulation and analyser to shutdown).