ENERGIZE – Glovebox Standard Operation Procedure

The glovebox at the future ENERGIZE beamline location is an MBraun UniLab Plus SP system, with N2-atmosphere. It provides a water- and oxygen-free environment, allowing for safe manipulation of air-sensitive materials. The ENERGIZE glovebox will mainly be a support for the ENERGIZE beamline, for sample mounting in an inert environment. It also has an integrated spin coater, in the event that sample preparation from solution is necessary.

Although mainly assigned to the ENERGIZE beamline, its use is open to all BESSY users provided they undergo a short introduction and comply with the guidelines given in this document and agreed upon before use.

The use of the glovebox must be scheduled beforehand, even for those who have had the introduction previously. In order to book it, please contact sample environment department.

• General Guidelines

Only users who have undergone the appropriate training and have booked the glovebox are allowed to use it.

The use of the glovebox (type of samples, procedures, solvent use, etc.) must be discussed beforehand. In particular, all samples and materials requiring special handling (toxicity, contamination concerns, etc.) and its specificity must be disclosed to the sample environment group before use. All the requirements must be met. Failure to comply with the rules will lead to a ban from using the glovebox – either temporarily or permanently.

• Overall rules of operation in the glovebox

To preserve the inert atmosphere in the glovebox, any item to be introduced in the system must be as free from oxygen and moisture as possible.

Any procedure (including introducing or removing materials from the glovebox) or significant event must be logged in the logbook, including name of the user and date.

Due to the limited space available in the glovebox, no storage of materials is allowed. Each user should bring a box properly labelled to store his/her materials during the time of the experimental run. These must be removed from the glovebox as soon as they are not needed, or as soon as the booked time is over. Unlabeled material will be removed from the glovebox and disposed of without notice.

All waste must be removed from the glovebox when you are done for the day, and the glovebox cleaned and tidied up.

Always remove watches, bracelets and rings before using the gloves. Your nails should be cut short and splinter-free.

Clean gloves must be worn before inserting hands in the glovebox gloves.

The use of long sleeve clothing (or inner gloves with sleeves) is mandatory.

The use of clean gloves over the main glovebox gloves is mandatory when working with solvents, and advised in any other case.

The placement of aluminum foil in the work area to collect spillages is advised and must be collected at the end of the experimental run.

The use of sharps is discouraged and will only be allowed on a case-by-case fashion. When allowed, sharps need to be clearly marked and removed from the glovebox as soon as possible. The use of cut-resistant gloves is recommended – ask the team if any are available in case you need to use these type of gloves.

• Glovebox atmosphere

Before starting any work in the glovebox, check the sensor readings for the glovebox pressure, oxygen- and water-levels, and the pressure in the main N2-bottle. These values should be logged in the logbook at the beginning and at the end of your experimental run, each day.

The O2- and H2O-levels should normally be well under 5 ppm. In case any of these are over 5 ppm, log it in the logbook and inform a member of the sample environment group immediately.

If the pressure in the Nitrogen bottle reaches 15 bar, log it in the logbook and inform a member of the sample environment group so it can be replaced as soon as possible.

The standard working pressure in the glovebox is set to 1 – 4 mbar. The inert gas pressure inside the glovebox should always be slightly above atmospheric (gloves in extended position), to avoid back-sucking air into the chamber. Although the glovebox regulates the pressure automatically, it can also be readjusted manually, using the foot pedal lying on the floor. If the pressure is too high, press the left pedal button to decrease it; if it is too low, press the right pedal button to increase it.

The glovebox operating parameters should not be modified by the users.

• Allowed samples

The glovebox is intended primarily for sample mounting, not sample preparation.

In the cases where sample preparation cannot be avoided, the details of the procedure must be discussed beforehand with the sample environment group.

Nevertheless, the following rules apply:

Glassware should be dried in an oven for a few hours before being introduced in the glovebox.

All vessels brought into the glovebox must be in an inert atmosphere (Nitrogen- or Argon-filled) or be evacuated before being introduced. Make sure that all empty containers are open.

Sample containers that have been kept in the cold must be allowed to reach room temperature before being brought into the glovebox to avoid introducing condensed moisture.

Solid materials and powders should be in an inert atmosphere, ideally in a new pre-packaged bottle straight after purchase. If that is not the case and the material is non-volatile, stretch a piece of parafilm over the opening of the bottle and poke a few holes in it to allow gas flow in and out of the bottle. Alternatively, place a paper tissue over the opening of the bottle and secure it with a rubber band. Introduce open in the glovebox, following the normal procedure described below (Glovebox operation section).

Solvent use should be minimized. Their use must be discussed beforehand, and their introduction to the glovebox agreed upon then. In general, any liquids to be introduced in the chamber should be anhydrous and degassed.

No samples or other materials are allowed inside the glovebox beyond the experiment run booked.

All trash and material that is no longer necessary must be removed from the glovebox at the end of the day, and the glovebox cleaned and tied up.

General materials like kimwipes, aluminum foil, gloves, etc. are available inside the glovebox. If the stock is running low, please inform the sample environment group. Do not introduce these items on your own. Only authorized staff is allowed to do so.

• Glovebox Operation

The ENERGIZE glovebox has one set of gloves and two antechambers – a small one and a large one. The thick butyl gloves allow the users to manipulate their samples and perform general laboratory tasks through the glass walls, without breaking the inert environment. Any necessary material is introduced in the main glovebox chamber through the antechambers, after a set of evacuation/flushing cycles with nitrogen that ensures it is an oxygen- and water-free environment.

Both antechambers should always be kept in vacuum when not in active use. This means that after introducing your materials you should always evacuate the antechamber, even if you think you will only take 5 minutes and need to come out again. Better to waste nitrogen than to risk opening the main chamber to air.

Gloves

The gloves are the weakest point in the glovebox, and are prone to small punctures, tears or cracks that can compromise the inert atmosphere of the main glovebox chamber. Before and after each use, inspect the gloves for holes or cracks in the rubber, and inform the sample environment group immediately in case you find any. There will be a glove port cover available inside the chamber for large leaks and emergency situations.

Always use long sleeves when working at the glovebox. Remove watches, bracelets and rings before using the gloves. Your nails should be cut short and splinter-free.

Before inserting your hands in the glovebox gloves, put on a clean pair of gloves. For extended periods of work and during warm days, you may use baby powder inside the clean gloves to minimize discomfort. You can find one bottle of baby powder on top of the glovebox or inside the cabinet.

Make use of the pressure pedal as you enter with your arms in the gloves and glovebox, lowering the pressure just enough not to feel too much resistance. Place the fingers of your non-dominant hand in the glove on the outside and then the ones of your dominant hand. Introduce one arm at a time, in a gentle and smooth movement, as you extend the arm through the glovebox window. Be cautious not to pull the gloves off the glove ports.

When removing your arms, make a fist with your hands, and remove the gloves gently. Making a fist helps in maintaining the fingers in the proper position for the next user. Do not pull out violently. Adjust the pressure with the pressure pedal if necessary.

Small antechamber

Minimize danger of contamination by grouping the material you need to introduce in the glovebox in as few introductions as possible. Also minimize the time the antechamber is open to air by having your material ready.

Never open the inner and outer antechamber doors simultaneously.

To introduce items in the glovebox:

1. Make sure the inner door of the antechamber is closed and that the antechamber is evacuated;

2. Turn manual valve slowly to the refill position and fill the antechamber with nitrogen;

3. Close the valve once the antechamber is full (vertical position);

4. Open the outer door, pull tray out and place your items on it, push tray in and close the outer door. Make sure all containers in your set of items are open;

5. Place the antechamber under vacuum by turning the manual valve slowly to the evacuate position, and leave it under vacuum for at least 3 minutes (Note: powders and light items may fly around in the chamber if not evacuated slowly);

6. Close the manual valve and turn it slowly to the refill position, filling it again with nitrogen;

7. Repeat steps 5 to 6 two more times;

8. Close manual valve (vertical position);

9. Put your hands in the main glovebox hands, reach for the inner door of the antechamber, open it and remove your things.

10. Close the antechamber door and evacuate the antechamber before continuing with your work.

To remove items from the glovebox:

1. Make sure the inner door of the antechamber is closed and that the antechamber is evacuated;

2. Turn manual valve slowly to the refill position and fill the antechamber with nitrogen;

3. Close the valve once the antechamber is full (vertical position);

4. Open the inner door, pull tray out and place your items on it, push tray in and close the inner door again tightly;

5. Open the outer door of the antechamber, remove your things to air, and close the door again;

6. Turn manual valve slowly to the evacuate position;

7. Leave the antechamber under vacuum, with the valve in the closed position.

Large antechamber

The use of the large antechamber is restricted. Please talk to someone from the sample environment group if you need to use it.