

Transferring Volatiles into the Glovebox

SOP for Introducing Sealed Containers of Liquids or Pastes into the Glovebox

Paul Davis, Sophia Mitchell, and Jesse Schimpf

Background

To ensure the continued integrity of the controlled atmosphere (Ar with <0.1 ppm H_2O and O_2) inside the glovebox as well as avoid potentially damaging the gas purifier and other glovebox atmosphere scrubber assemblies, it is crucial to avoid introduction of oxygen, water, or harmful volatile compounds. Likewise, while nitrogen gas (N_2) is typically thought of as inert, it can react with metal ion battery electrodes, negatively impacting experiments. Thus it is imperative to always check for chemical compatibility prior to introducing a new compound or material into the glovebox, as well as to eliminate any adsorbed water or trapped oxygen or nitrogen (the primary components of air) on materials or in containers. The following standard operating procedure (SOP) details how to transfer sealed containers of volatiles (e.g., liquids or pastes) into the glovebox without introducing oxygen, water, or nitrogen, or conversely losing all of the desired volatile(s) during the antechamber evacuation cycle(s).

Procedure

1. Attach the inert gas regulator and Teflon gun assembly from the compressed UHP N_2 tank to the spare full Ar tank.
2. Use the gun assembly from the UHP N_2 tank to remove the air (in particular, O_2 , H_2O , and N_2) from the headspace of the bottle containing the volatile(s) to be transported into the glovebox by replacing the air with Ar (i.e., filling the headspace with Ar from the gun).
 - a. After flowing Ar through the bottle for approximately 30 seconds to displace as much air as possible, rapidly close the lid of the bottle to minimize reintroduction of air via diffusion.
3. Place the closed/sealed bottle (filled with Ar) inside a large, dry, open plastic Ziplok bag.
4. Place the bag containing the bottle in the glovebox's mini-antechamber.
 - a. Note that you may have to put the Ar-filled Ziplok into the large (full-size) antechamber if the bag or bottle is too large for the mini-antechamber, but the mini-antechamber is preferable as it will pump down more quickly and can achieve a higher vacuum (i.e., lower final pressure/gaseous contaminant level).
5. Run a manual antechamber cycle.
 - a. Evacuate to 1 mbar.
 - i. Do not let the pressure drop below 0.5 mbar, as that will cause the turbomolecular pump to kick on!

- ii. If you are using the large antechamber, pump until the pressure gauge dial reading stops changing, then continue pumping for a few more minutes.
 - b. Refill the chamber to atmosphere.
 - c. Repeat this cycle 3 times. This should (via serial dilution) remove almost all the residual air (i.e., O₂, H₂O, and N₂) from the antechamber without (hopefully) compromising the seal on the bottle and causing the volatile component(s) to evaporate (i.e., removing the liquid or drying out the paste).
6. Bring the open Ziplok bag and sealed/closed bottle into the glovebox.
7. Seal the Ziplok bag, attempting to fill it with as much Ar from the glovebox atmosphere as possible.
8. With the bottle still inside the Ar-filled, sealed Ziplok bag, open the cap on the bottle.
9. Allow the bottle headspace to equilibrate with the Ar atmosphere inside the sealed Ziplok bag for 5-10 minutes.
 - a. Note that this step can be performed either inside the glovebox or in the antechamber. If you choose the latter, make sure the bottle remains upright so that the liquid or paste doesn't spill out.
10. Close/reseal the bottle (still inside the sealed Ziplok bag).
11. Put the closed bottle housed inside the sealed Ziplok bag into the mini-antechamber and bring the bag out to atmosphere to release any residual air (i.e., O₂, H₂O, and N₂) in the bag that came from the bottle headspace.
 - a. You may need to put the Ar-filled Ziplok into the large antechamber if the bag's volume is too large to place in the mini-antechamber. Do not attempt to squeeze or pop the bag.
12. Quickly put the sealed bottle back into the mini-antechamber and run another manual cycle (same parameters as in Step 5 above).
13. The bottle/container should now be filled with Ar and can safely be brought into and opened/used inside the glovebox without causing a spike in either O₂ or H₂O levels.