2: Cycling onto the Schlenk Line

Glassware used for the manipulation and preparation of air- and moisture-sensitive compounds must be rigorously dried and free of air prior to use. Schlenk flasks and items are typically stored in ovens above 100 ºC (for a few hours or overnight) to remove any residual solvent or adsorbed water.

Attaching a new Schlenk flask or reaction vessel to the Schlenk line

Step 1

Remove a Schlenk flask, magnetic stir bar, ground glass stopper and stopcock from the oven using heat resistant gloves.

Schlenk flask, stopcock, stopper and stir bar.

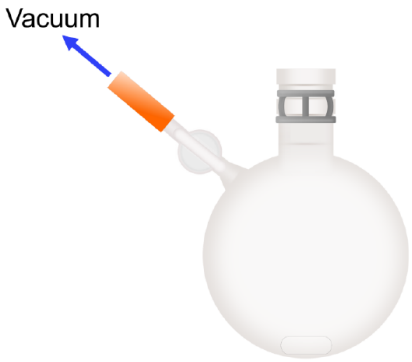
Step 2

The stopcock and stopper are greased (using a length of silicone, hydrocarbon or polyfluorinated based lubricant) and inserted into the corresponding ground glass joints with twisting to ensure a smooth and uniform seal that is free of bubbles or streaks. Only two thirds of the stopper are greased to prevent excess grease from contaminating the inside of the flask. Ensure that the grease does not block the gas inlet/outlet hole on the stopcock. Appropriate nuts, O-rings, washers and clips can be added to secure the stopcock and stopper in place.

Greased and assembled Schlenk flask.

Step 3

The assembled Schlenk flask is then connected to the Schlenk line hosing and evacuated under vacuum whilst it cools to room temperature. After a suitable amount of time (depending on the size of the flask, ~5 minutes for a 100 mL vessel), the Schlenk flask is slowly backfilled with inert gas. This corresponds to a single ‘cycle’, and this is repeated two more times to ensure complete removal of air and moisture from the reaction vessel.



Evacuating a Schlenk flask under vacuum.

Cycling sealed vessels already under an inert atmosphere:

For ampoules and flasks that are already sealed under an atmosphere of inert gas, three vacuum/inert gas cycles are required to evacuate the air within the Schlenk line hosing and flask side arm. Shorter cycles (30-60 seconds) are typically sufficient for these purposes.

The process of performing numerous vacuum/inert gas cycles is akin to performing repeated extractions of an aqueous phase with organic solvents during a typical reaction work-up. Assuming that a 100 mL reaction flask contains approximately 1 mmol of O_2 at room temperature and atmospheric–pressure (~1000 mbar), then an initial vacuum cycle down to 0.1 mbar will reduce the quantity of O_2 down to 1 x 10^{-4} mmol of O_2. Backfilling with inert gas and re-evacuating the flask down to 0.1 mbar again reduces the O_2 quantity down to 1 x 10^{-8} mmol, and a third vacuum/inert-gas cycle further lowers this to 1 x 10^{-12} mmol of O_2. This is several orders of magnitude lower than performing a single but longer evacuation down to 0.001 mbar, for example, and illustrates that even poor performing vacuum pumps can be sufficient to reach low partial pressures of O_2 after repeated vacuum/inert gas cycles.
