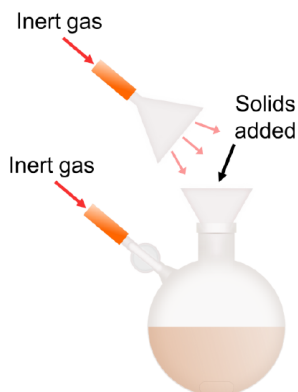


11: Addition of Solids

It may be more convenient (or even necessary) to add solid reagents directly to the reaction flask instead of first dissolving it in a separate Schlenk flask and performing a cannula transfer.

For less sensitive reactions involving air- and moisture-stable solids, it is often sufficient to simply add the solid through a powder funnel (to avoid it sticking to the greased ground glass joint) under a positive flow of inert gas. An inert gas blanket can also be employed – this is achieved by inserting the stem of an inverted funnel into the Schlenk line hosing and flushing inert gas over the Schlenk flask during the addition of the solid. This method works best when using argon as an inert gas since it is denser than both air and N_2 . This method is particularly useful for the portion-wise addition of air- and moisture-stable solids.



Using an inert gas blanket to add solids.

The addition of sensitive solid reagents can be achieved by using a solid addition tube – these are similar to test tubes but typically contain a bend ($\sim 45^\circ$) and a ground glass male joint with can be sealed with a Schlenk cap, small round bottom flask or a rubber septum. Solid addition tubes may also contain a gas inlet sidearm with a ground glass stopcock or Teflon tap to control the inert gas atmosphere.

Step 1

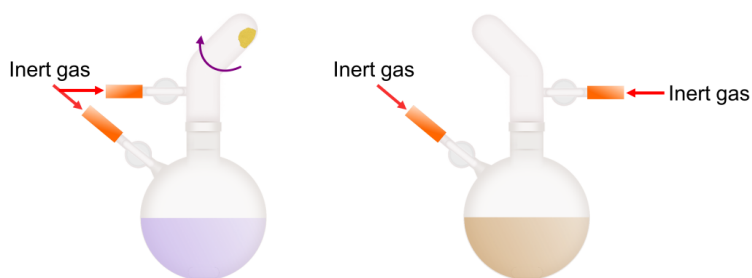
The solid reagent is first added to the solid addition tube inside a glovebox, and this can then be cyclized onto the Schlenk line.



Cycling the solid addition tube onto the Schlenk line.

Step 2

Under a positive flow of inert gas, the stopper on the Schlenk flask is removed and the Schlenk cap is quickly removed from the solid addition tube to allow it to be connected to the Schlenk flask. Rotating the solid addition tube and gently agitating it will encourage the solid to enter the reaction flask. This method is best suited for the addition of crystalline solids or salts that will not stick to the solid addition tube.



Before and after addition of the solids.

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