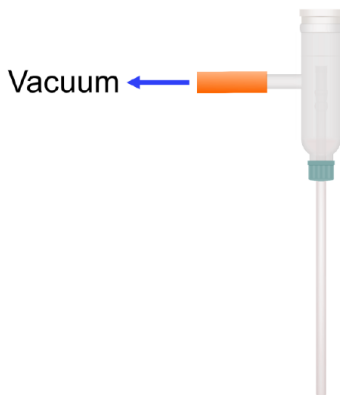


6.1: Preparing NMR Samples on a Schlenk Line

Air- and moisture-sensitive samples to be analysed by NMR spectroscopy can be readily prepared using Schlenk line techniques. NMR tubes used for sensitive samples typically contain a screw-thread Teflon tap (i.e. J. Young's) which ensures an airtight seal to prevent contamination by atmospheric air or moisture. Although it is often more convenient to prepare NMR samples inside of a glovebox, it may be desirable to add the deuterated solvent on a Schlenk line (to minimise solvent use in a glovebox or prevent contamination by other volatiles in the glovebox atmosphere) or to directly take an aliquot of the reaction mixture.

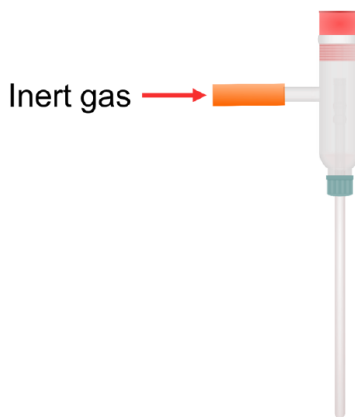
Preparing an NMR sample to analyse a reaction mixture

Step 1: Cycle a J. Young's NMR tube onto the Schlenk line with a minimum of three vacuum/inert gas cycles using an appropriate NMR tube adapter. A thermometer adapter or a rubber septum with a 5 mm hole at the bottom of the adapter provide a sufficiently air-tight seal.



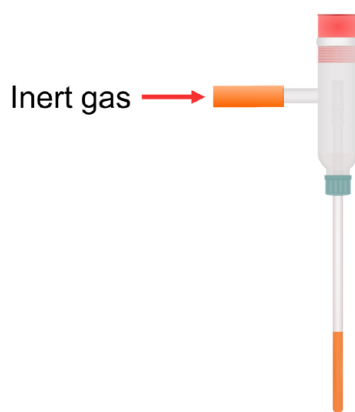
Cycling a J. Young's NMR tube onto the Schlenk line.

Step 2: Replace the glass stopper with a rubber septum under a positive pressure of inert gas. Insert a bleed needle for 5-15 seconds to purge out any air that enters the system.



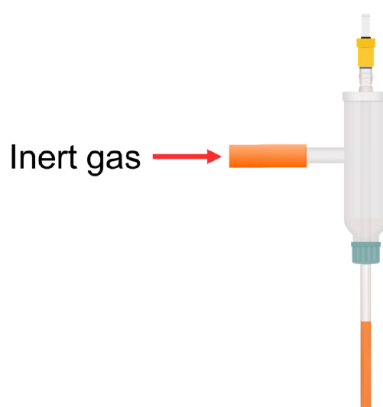
Fitting the rubber septum to the NMR tube adapter.

Step 3: Add an aliquot of the reaction mixture (approximately 0.5-0.6 mL) to the NMR tube via [cannula transfer](#) (using a suitably gauged cannula) or with a syringe and needle.



Adding an aliquot of the crude reaction mixture.

Step 4: Under a positive pressure of inert gas, secure the Teflon valve onto the J. Young's NMR tube. Close the stopcock or Teflon tap supplying the inert gas from the Schlenk line into the NMR tube adapter.

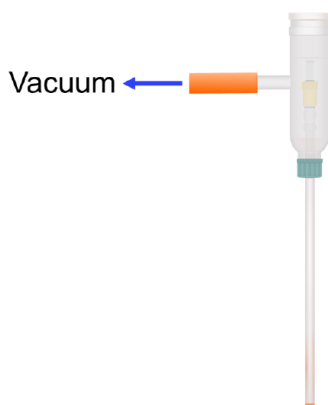


Sealing the NMR tube under inert gas.

The *protio*-solvent in the NMR tube can now be removed under vacuum and the sample later redissolved in an appropriate deuterated solvent. Alternatively, the NMR sample can be run directly provided that the NMR spectrometer is setup for *protio*-solvents – this is particularly useful for heteronuclei such as ^{31}P NMR spectroscopy.

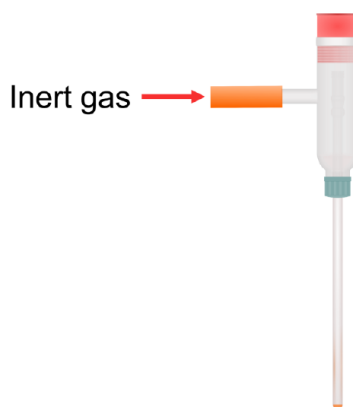
Adding solvent or liquids to a sealed J. Young's NMR tube on the Schlenk line

Step 1: Cycle the sealed J. Young's NMR tube onto the Schlenk line using an appropriate adapter.



Cycling a sealed J. Young's NMR tube onto the Schlenk line.

Step 2: Under a positive pressure of inert gas, remove the glass stopper, raise the NMR tube and remove the J. Young's Teflon valve. Quickly lower the NMR tube and seal the adapter with a rubber septum. Insert a bleed needle through the septum for 10-15 seconds to purge out any air that is introduced into the system.



Removing the Teflon valve under inert gas.

Step 3: Add the desired solvent or liquid reagent to the NMR tube *via* [cannula transfer](#) or needle and syringe.

Step 4: Under a positive pressure of inert gas, secure the Teflon valve onto the J. Young's NMR tube. Close the stopcock or Teflon tap supplying the inert gas from the Schlenk line into the NMR tube adapter.

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