

6.2: Removing Solvent from NMR tubes

Solvent can be removed directly from NMR tubes under vacuum by using the tapered glass adapters that are supplied with J. Young's NMR tubes.

Step 1

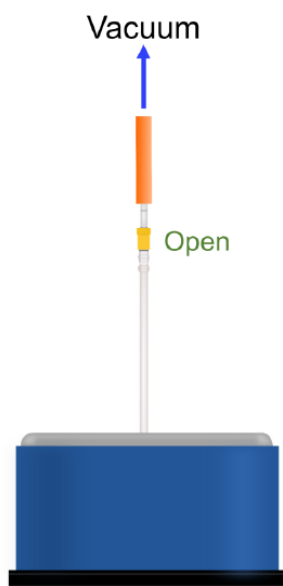
Attach the J. Young's NMR tube directly to the Schlenk line hosing using the tapered glass adapter and place under vacuum.



Attaching the NMR tube to the Schlenk line hosing.

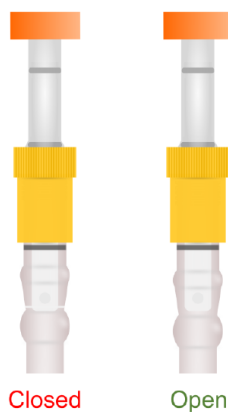
Step 2

The contents of the NMR tube are cooled (or frozen) in a dry ice/acetone bath (-78 °C) or a Dewar of liquid nitrogen. Once the solvent is frozen, open the Teflon valve carefully by twisting it anti-clockwise, and evacuate the headspace for 30-60 seconds.



Evacuating the headspace of the NMR tube.

The J. Young's valves are hollow, allowing the NMR tube to be opened or closed to vacuum or inert gas whilst connected to the Schlenk line hosing.



Closed and open positions of a J. Young's NMR valve.

Step 3

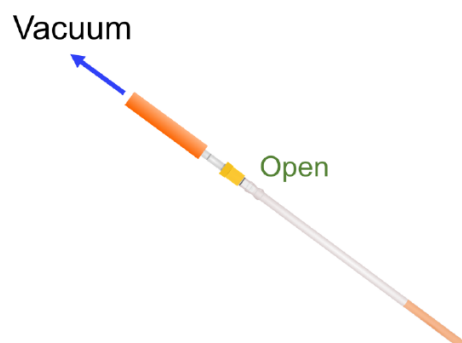
Close the Teflon valve and remove the NMR tube from the cooling bath. Carefully thaw the solvent from the top down to the bottom by rubbing the tube between your thumb and fingers. This degasses the solvent which helps prevent bumping whilst under dynamic vacuum.



Thawing the contents of the NMR tube under static vacuum.

Step 4

Repeat step 2 then remove the NMR tube from the cooling bath and thaw the solvent with the Teflon valve open. As the NMR tube warms to room temperature, the solvent will slowly evaporate. Holding the NMR tube at an angle with gentle agitation helps to increase the surface area to facilitate evaporation of the solvent. It may be necessary to close the Teflon tap and repeat this process if the solvent removal becomes uncontrolled.



Removing solvent from an NMR tube under dynamic vacuum.

Step 5

Keep the NMR tube under vacuum for 5 or so minutes to remove all traces of solvent and volatile material. Backfill the NMR tube with inert gas and then close the Teflon valve. The NMR tube is now ready to be transferred into a glovebox or cycled back onto the Schlenk line for further manipulations.



NMR tube after removal of solvent.

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