7.1: Static Vacuum Distillation

Static vacuum distillations (also called vacuum transfers) are frequently employed for the purification and/or drying of small volumes of solvents or liquid reagents with relatively low boiling points (<150 °C). Static vacuum distillations often employ custom-made greaseless vessels and distillation bridges which prevents grease contamination in the purified distillate.

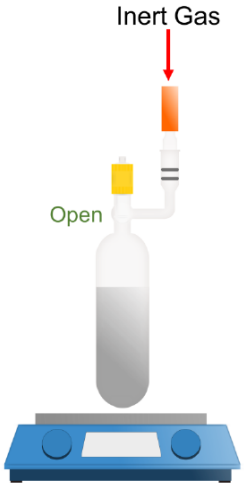
Step 1

An oven-dried Teflon tapped ampoule with a 90° side-arm, equipped with a stir bar, is cycled onto the Schlenk Line using a suitable O-ring or ground-glass adapter.

Cycling a Teflon tapped ampoule onto the Schlenk line.

Step 2

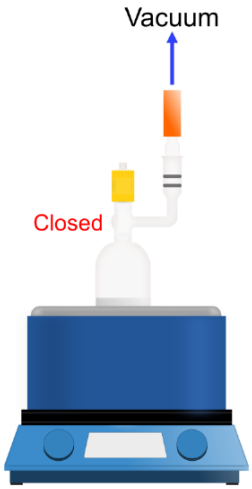
The ampoule is backfilled with inert gas and then the desiccant is added followed by the solvent (or liquid reagent); this can be introduced via syringe or cannula transfer, or simply poured into the flask with a funnel under a flow of inert gas. The contents are then stirred overnight under an inert gas atmosphere: the flask can be sealed or periodically opened to the Schlenk line to release H_2 build-up (from the reaction of the desiccant with water or protic impurities).



Drying the solvent or liquid reagent over a dessicant.

Step 3

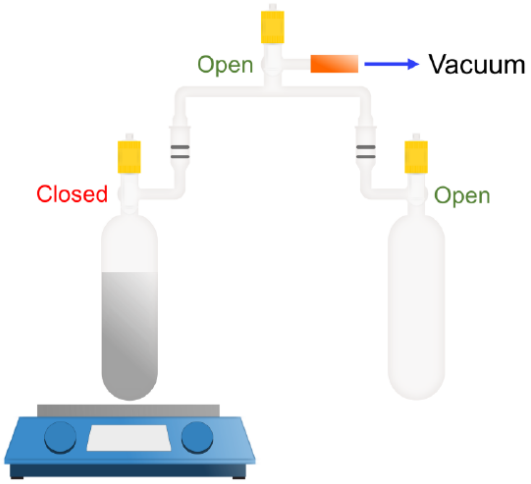
The Teflon tap is closed, and the ampoule is slowly submerged in a -78 °C dry-ice/acetone bath. Once completely frozen, the headspace is evacuated under dynamic vacuum for 5 minutes. The Teflon tap is then closed, and the ampoule is removed from the cooling bath to slowly thaw to room temperature. This corresponds to one freeze-pump-thaw cycle, which is then repeated once more.



Performing a freeze-pump-thaw to degas the contents of the ampoule.

Step 4

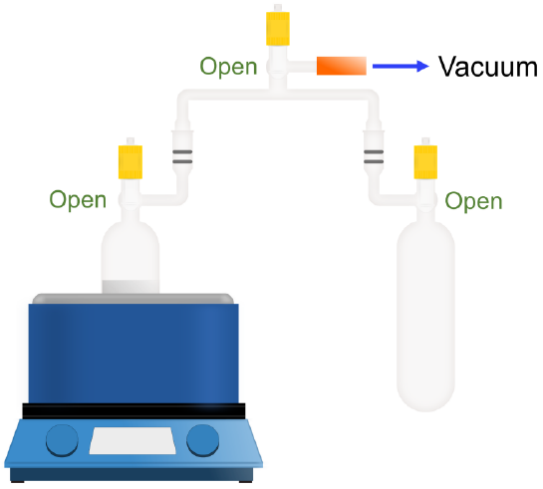
The ampoule is then connected to a greaseless distillation bridge (this may be built directly into the Schlenk or high vacuum line, or be a separate unit that can be connected to the Schlenk line hosing), and a second receiving ampoule is connected to the bridge and evacuated under vacuum. Oven-dried glass wool can be placed within the distillation bridge or ampoule side-arm to prevent solid desiccant from accidentally contaminating the purified distillate.



Connecting a receiving ampoule to the distillation bridge.

Step 5

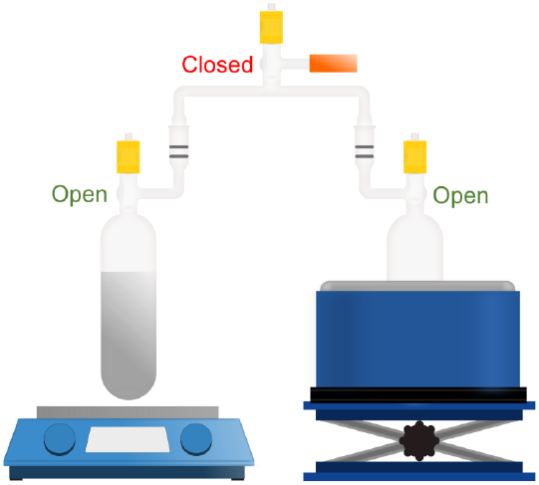
The ampoule containing the liquid is cooled to -78 °C using a dry-ice/acetone bath, and then evacuated under vacuum. Liquid nitrogen cooling baths should be avoided where possible for vacuum transfers as this presents a significant risk of liquid oxygen condensation. Performing this third freeze and pump cycle whilst attached to the distillation bridge ensures that the best possible vacuum is established within the distillation apparatus. A manometer is beneficial here to confirm a suitable vacuum is established prior to beginning the distillation.



Establishing the static vacuum.

Step 6

The Teflon tap on the distillation bridge is closed, then the transfer ampoule is removed from the -78 °C cooling bath whilst the receiving ampoule is submerged in the -78 °C cooling bath . As the liquid slowly thaws, it will begin to evaporate under the reduced pressure and condense in the receiving flask at the lower temperature.

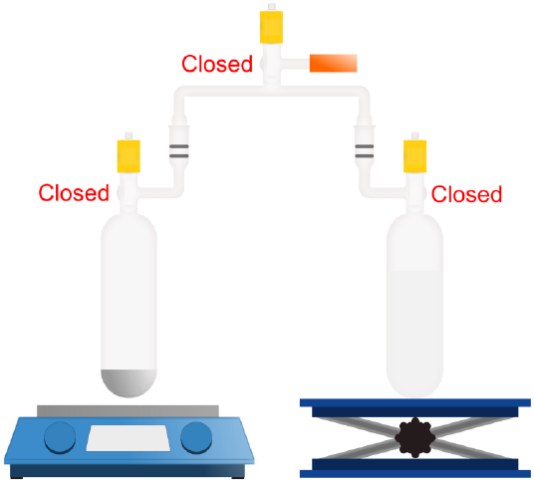


Performing a static vacuum distillation.

It may be necessary to refresh the static vacuum periodically by closing the Teflon tap on the transfer ampoule, then opening the Teflon tap on the distillation bridge to dynamic vacuum. After several minutes, or until the manometer reading is sufficiently low, the Teflon tap on the distillation bridge is closed and the Teflon tap on the transfer ampoule is slowly opened to the continue the distillation.

Step 7

Once the distillation is complete, the Teflon taps on both ampoules are closed and the receiving flask is removed from the cooling bath. The liquid in the receiving flask is allowed to thaw before being backfilled with inert gas (depending on the setup this may need to be first disconnected from the distillation bridge and then cycled back onto the Schlenk line). The freshly distilled liquid can now be transferred to a suitable flask for storage. The transfer ampoule is also cycled onto the Schlenk line and backfilled with inert gas, and then quenched with iso-propanol/toluene to safely destroy the excess desiccant.



Completed static vacuum distillation.
