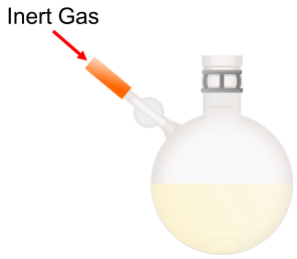
7.2: Dynamic Vacuum Distillation

Dynamic vacuum distillations are routinely employed to purify high-boiling liquids (> 150 °C) and some low-melting solids. This method is well suited for commercially available reagents or compounds prepared on a large scale in the laboratory in which the (often known) impurities are non-volatile and so remain behind after distillation.

Step 1

The impure material is transferred to a suitable Schlenk flask equipped with a magnetic stir bar. For commercially available reagents to be purified in bulk, this can be added to a Schlenk flask (that has already been cycled onto the Schlenk line) under a positive pressure of inert gas. For “in-house” prepared compounds, the crude material will typically remain in the Schlenk flask after removing the solvent and volatiles *in vacuo*.



Crude material in a Schlenk flask.

Step 2

A Schlenk flask, distillation bridge and Schlenk cap are greased, assembled and cycled onto the Schlenk line. Note: The style of distillation bridge (single-piece) illustrated is designed specifically to purify high-boiling liquids under high-vacuum. This differs from a typical distillation setup with a distillation head, thermometer adapter, and water-cooled condenser.

Cycling the distillation bridge and receiving flask onto the Schlenk line.

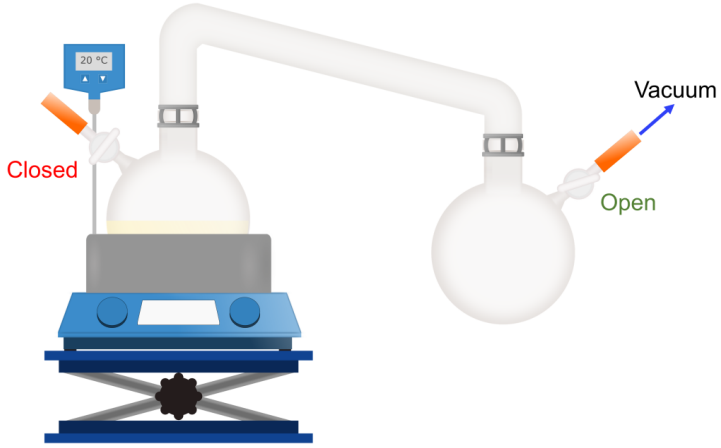
Step 3

Once the receiving Schlenk flask and distillation bridge has been cycled onto the line and back-filled with inert gas, it is connected to the Schlenk flask containing the crude material. This may require a brief helping hand to remove clips and stoppers. Ensure that inert gas is flowing into both flasks during this process to minimise exposure to atmospheric air and moisture.

Setup prior to distillation.

Step 4

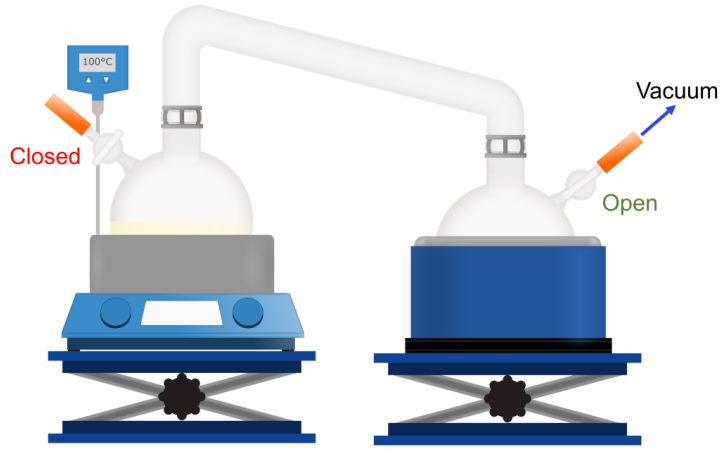
The stopcocks on both Schlenk flasks are closed and the distillation flask is lowered into a suitable heating mantle or oil bath. With stirring, the stopcock on the receiving Schlenk flask is slowly and carefully opened to vacuum. This serves to degas the crude material and remove any residual solvent or volatile impurities. *Note: The bulk material should be sufficiently high-boiling so that it does not evaporate at ambient temperatures whilst under vacuum. An external liquid nitrogen trap can be used between the receiving flask and Schlenk line to condense any volatile compounds.*



Evacuating the assembly prior to heating.

Step 5

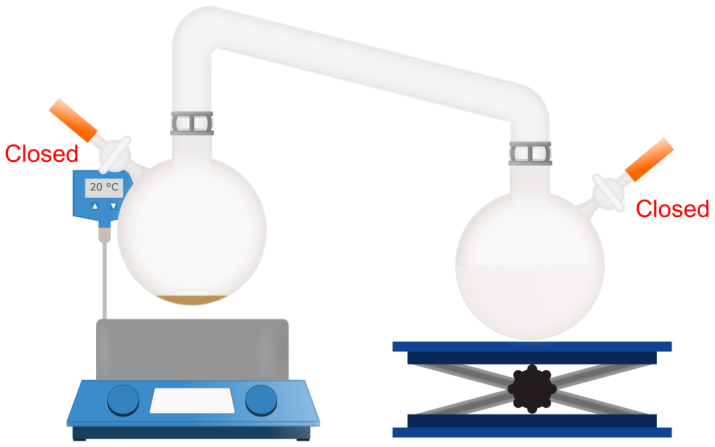
Once a good vacuum (*i.e.* low pressure) has been established within the distillation setup, and the crude material is fully degassed, the temperature on the heating mantle can slowly be increased. Since the crude material does not evaporate at ambient temperature, an ice bath is generally sufficient to cool the receiving flask and condense the distillate, however a dry-ice/acetone bath can also be used. During the distillation, it may be necessary to insulate the flask and part of the bridge with aluminium foil, or to briefly heat the glassware with a heat-gun.



Performing a dynamic vacuum distillation.

Step 6

Once the distillation is complete, the stopcock is closed on the receiving flask. The heating mantle is lowered to allow the distillation flask to cool to ambient temperature, and the cooling bath is removed from the receiving flask to allow it to thaw or warm to ambient temperature.



Completed distillation.

Step 7

When the distillation apparatus is at room temperature, the system is slowly backfilled with inert gas. If an external trap was used, it is necessary to disconnect this first and cycle the receiving flask back onto the Schlenk line.

Step 8

Under a positive pressure of inert gas, the distillation bridge can be removed from the receiving flask and replaced with a clean, greased ground-glass stopper. The purified material can now be transferred to a suitable ampoule for storage *via* cannula transfer, or used directly for further manipulations.

Hints and tips

- An approximate distillation temperature can be calculated using the known boiling point of the compound (at ambient pressure) and the pressure within the Schlenk line (if a manometer is being used).
- For complex mixtures of species to be separated by vacuum distillation, a more elaborate setup containing a vigreux column, a thermometer adapter, and a ‘pig’ receiver is generally required to collect multiple fractions.