

## 12: Refluxing Under an Inert Atmosphere

### Method A

#### Step 1

Cycle a reflux condenser onto the Schlenk line. Attach a gas inlet adapter to the top of the condenser, and a Schlenk cap or small round bottom flask to the bottom of the condenser, greasing the male joints and ensuring a uniform seal. Evacuate under vacuum.



Cycling a reflux condenser onto the Schlenk line.

#### Step 2

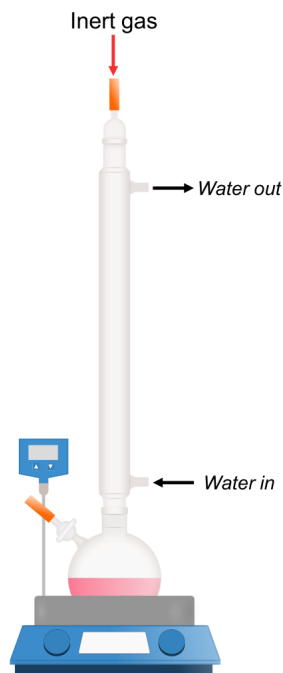
Once the reflux condenser has been cycled onto the Schlenk line, backfill with inert gas and attach to the Schlenk flask under a positive pressure of inert gas.



Attaching the condenser to the Schlenk flask.

### Step 3

Close the stopcock supplying inert gas to the Schlenk flask and lower the flask into a heating mantle or oil bath. Connect the water tubing to the reflux condenser and begin heating.



Refluxing under an inert atmosphere.

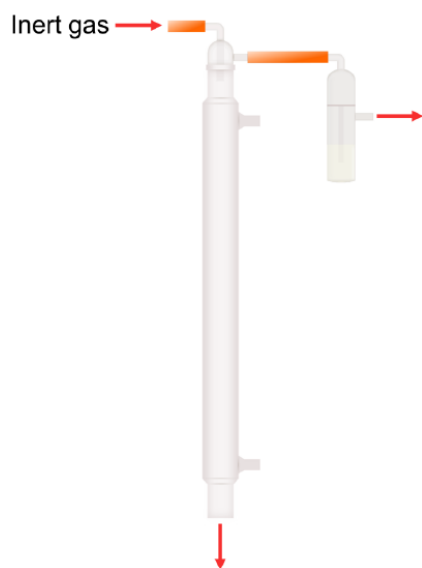
The inert gas flow rate, which can be monitored by the frequency of bubbling, can be lowered once the desired temperature is reached, but should be increased once the flask cools back to room temperature to prevent oil suck back. It is generally recommended to not perform other reactions on the Schlenk line during a reflux to prevent contamination with solvent vapours.

### Method B

If the reflux is expected to liberate nasty by-products that may contaminate the Schlenk line, then it is advised to use an external bubbler.

#### Step 1

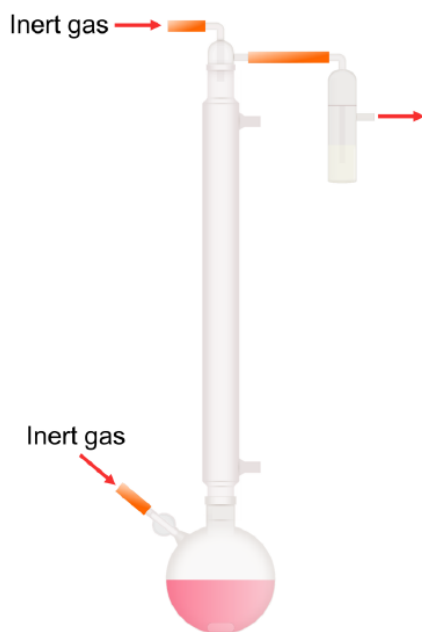
Assemble a reflux condenser and Dreschel head (gas inlet/outlet adapter), greasing the male joints and ensuring a uniform seal. Connect an external oil bubbler to the gas outlet and then attach the gas inlet to the Schlenk line hosing. Open to the inert gas and purge the glassware for 30-60 seconds.



Purging a reflux condenser equipped with an external bubbler.

### Step 2

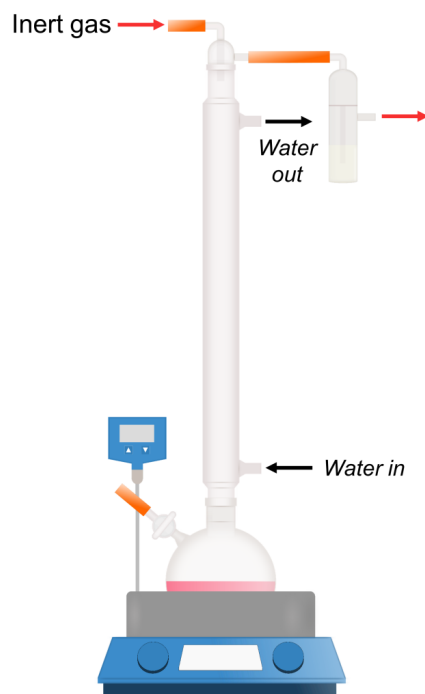
Attach the reflux condenser to the Schlenk flask under a positive pressure of inert gas. The external bubbler will begin bubbling at this point – adjust the gas flow so that it bubbles once every few seconds.



Attaching the condenser to the Schlenk flask.

### Step 3

Close the stopcock supplying inert gas to the Schlenk flask and lower the flask into a heating mantle or oil bath. Connect the water tubing to the reflux condenser and begin heating. It may be necessary to adjust the inert gas flow rate as the system approaches to the desired temperature.



Refluxing under an inert atmosphere with an external bubbler.

#### Step 4

After the desired duration of heating, cool the reaction mixture to room temperature. Increase the inert gas flow rate whilst cooling to prevent oil suck-back. Once cooled to room temperature, open the stopcock on the Schlenk flask and replace the reflux condenser with a greased stopper under a positive pressure of inert gas.

#### Method C

Reactions that require prolonged heating, and do not liberate gaseous by-products, can be heated directly in Teflon tapped ampoules without the need for a reflux condenser. Since this method does build up pressure within a sealed vessel, it is essential that the reaction vessel is suitable for such use. It is advised not to heat beyond the boiling point of the solvent unless specialist heavy-walled pressure vessels are used in tandem with a blast shield.

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