

Rotary evaporators:

A rotary evaporator is used to remove a solvent from a flask without having to heat that flask to the boiling point of the solvent at one atmosphere. This has the advantage that it is quicker and is less likely to cause thermal decomposition to the sample.

All the rotary evaporators are connected to a source of vacuum. Most are attached to a mechanical pump through a communal manifold. The vacuum should be turned on first and turned off last. The evaporators are water cooled, usually by a recirculating system that again links several rotovaps together. It is not necessary to have a very fast flow of cooling water, but lowering the temperature with ice in the reservoir helps the condensation of the solvent.

It is advisable to use one of the anti-splash vessels between the flask and the evaporator. This will prevent the previous user's material from being washed down into your flask, provided the central tube of the anti-splash vessel has been cleaned.

Before putting the sample flask onto the evaporator, close the tap for the air leak. This will help to prevent the flask falling off again! Hold the flask for a few seconds until the vacuum can retain the flask. To be sure, use one of the Keck clips provided (green = B24; blue = B19; yellow = B14). Check the clip for cracks, and discard if faulty. Refrain from filling a flask more than half-full. Not only is it more likely to splash, but with larger flasks, the weight may be too much for the vacuum to keep it attached. Never use a flask larger than 1 litre. You will not be the first person to attempt an extraction from the water bath!

Once the pressure in the flask has dropped, turn on the rotary motor, slowly at first and then up to maximum if this does not cause too much turbulence. The flask may also be partially immersed in a water bath set at an appropriate temperature. Remember that some compounds (particularly organometallics) may decompose if the temperature is too hot. With very volatile solvents (as in this experiment), a room temperature water bath is necessary just to prevent the evaporating solvent from cooling the flask too much. It is necessary to cool the solvent receiving flask in ice as otherwise the evaporation may preferentially take place from there, causing solvent vapour to be drawn into the mechanical pump.

When you are finished, stop the rotation and remove the water bath from around the flask. Break the vacuum by opening the air leak slowly. If you have a fine powder left, this may be sprayed around the inside of the glassware unless the pressure is slowly returned to one atmosphere. Gently remove your flask. Turn off the following:

- (i) the heat control of the water bath.
- (ii) the source of vacuum.
- (iii) the cooling water.

Empty the receiving flask into the appropriate solvent waste container (no-one else will know what solvent you used), and clean the inside of the anti-splash vessel.