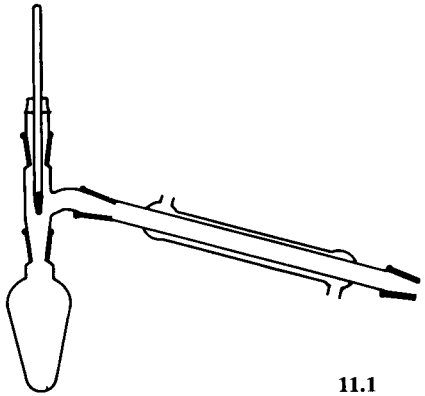
Chapter 11 Laboratory techniques

11.1 Distillation

Distillation is used to separate a liquid product of a chemical reaction from the reactants and by-products of the reaction.

The apparatus shown in diagram 11.1 provides a suitable arrangement. It is not always necessary to use a thermometer; this can be replaced by a stopper. Anti-bumping granules (pumice stone or pieces of broken porcelain) are added to the contents of the flask to ensure smooth boiling. The flask is heated steadily, either in a water bath, or an oil bath, or with a micro-burner. The latter can be either purchased or improvised by unscrewing the barrel from an ordinary bunsen burner. The distillate must come over slowly and the flask should never be boiled dry. The thermometer bulb must be placed as shown to ensure the temperature of the vapour leaving the flask is measured.



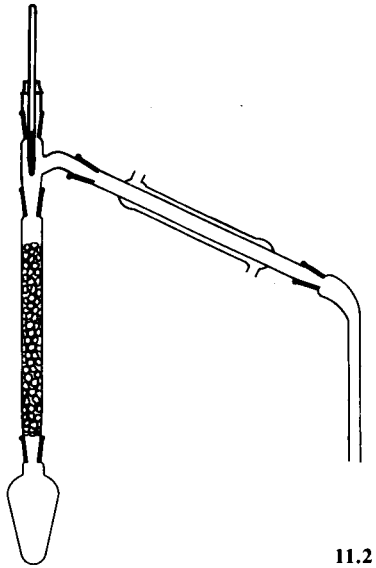
11.1

11.2 Fractional distillation

Fractional distillation is used to separate liquids which are miscible, and have different boiling points. When the mixture is boiled, vapours of both liquids go up the fractionating column. The column should be packed with glass beads or rings or a specially-made column can be used.

The vapour which reaches the top of the column is the one with the lowest boiling point (ie the most volatile). The liquids with higher boiling points condense in the column and fall back into the flask. The vapour which reaches the top of the column passes in to the water-cooled condenser and can be collected, as shown in diagram 11.2.

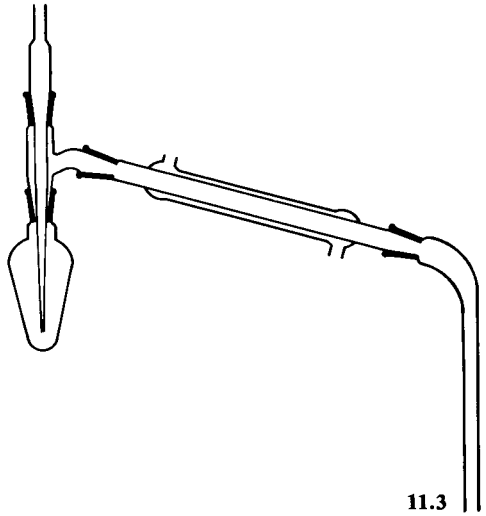
Further information on the theory of fractional distillation can be found in text-books of Physical Chemistry.



11.2

11.3 Steam distillation

Many water-insoluble compounds, both solid and liquid, may be purified by distillation in a current of steam, provided the compound is volatile and the impurities involatile. This enables high boiling point materials to be distilled considerably below their normal boiling point. A suitable arrangement is shown in diagram 11.3. Steam is passed through into the flask whilst heating the flask to prevent unnecessary condensation of steam in it. A tap funnel can be attached to the end of the Liebig condenser to act as a receiver if convenient to the next stage of the preparation, as where the distillate requires washing or separating.



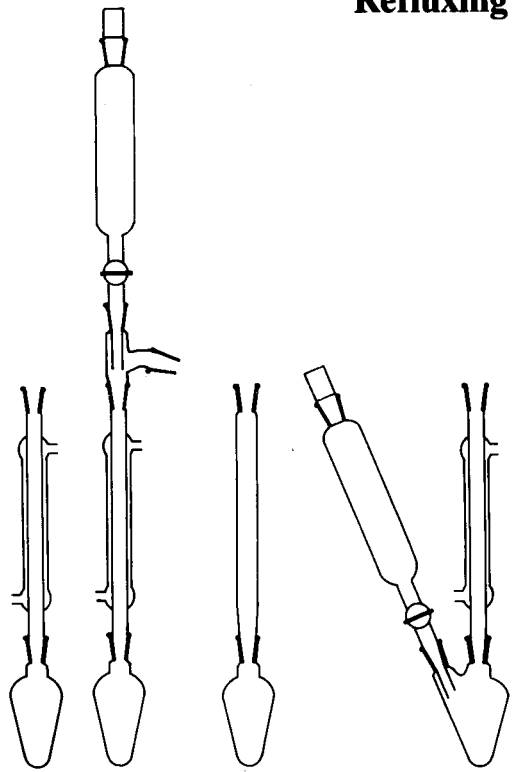
11.3

11.4 Refluxing

Refluxing is a technique used to keep a reaction mixture at its boiling point without loss of either solute or solvent.

For normal refluxing, a flask and condenser are sufficient, diagram 11.4a, and the flask should be gently heated by water bath, micro-burner, etc. When liquids with a boiling point in excess of 100°C are being refluxed it is advisable to use an air condenser, diagram 11.4d.

In diagrams 11.4b and 11.4c suitable arrangements are shown for "reflux with addition". In this technique a reagent is added to the contents of the flask which may initiate an exothermic reaction. The heat energy could result in the loss of a volatile reactant.



11.4a 11.4b 11.4c 11.4d