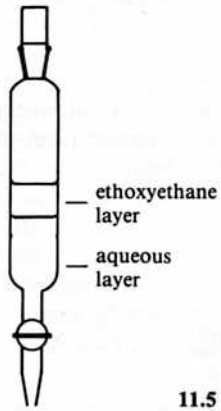
11.5 Solvent extraction

Solvent extraction is a technique used to separate a solid or liquid product from a reaction mixture. A solid or liquid which is soluble in two immiscible liquids will, if all three are mixed, distribute itself between the two liquids according to the Partition Law:

$$\frac{\text{concentration of solute in Solvent A}}{\text{concentration of solute in solvent B}} = \text{constant}$$

Usually, ethoxyethane and water are used as solvents. To the aqueous solution of the compound in a separating funnel, diagram 11.5, about half the solution volume of ethoxyethane is added. The funnel is shaken vigorously (hold the stopper in with the forefinger) and the pressure released frequently by inverting and opening the tap. No flames or other sources of ignition should be in the same room. After standing for a few minutes, two layers will form. The lower, water, layer should be run off and the ethoxyethane layer run off into a small bottle. Return the aqueous layer to the funnel and repeat with another portion of ethoxyethane. Dry the extracts with anhydrous magnesium sulphate. The ethoxyethane can be removed from the extract layer by distillation; heating should be on a pre-heated water bath.



11.5

Remember that multiple extraction with several small portions of ethoxyethane is more efficient than one extraction with a large portion.

11.6 Recrystallization

Recrystallization is the technique used to purify a compound which is contaminated with the starting materials or products of a side reaction. A solvent is required in which the compound to be purified is very soluble when hot but almost insoluble when cold. Insoluble impurities are removed by filtration when the solution is hot. Soluble impurities are removed by filtering off the product from the cold solvent.

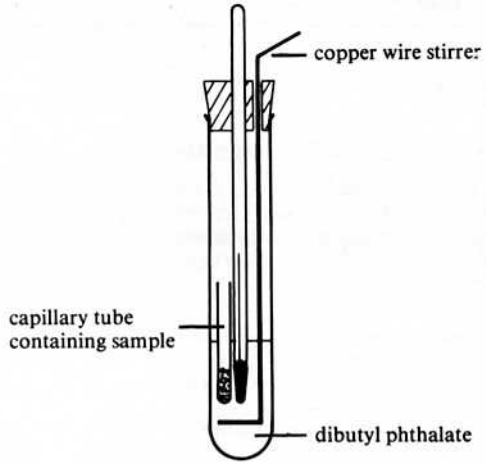
The crystals are dissolved in the minimum volume of hot solvent such as ethanol or propanone. Heating should be in a water bath or by refluxing. The hot solution is filtered, if insoluble impurities are present, by pouring into a filter funnel which has been pre-heated with hot solvent. Alternatively, if the volumes used are small, a centrifuge can be used and the still-hot liquid decanted into a clean test-tube.

The solution is allowed to cool and crystallize. The crystals are removed, preferably by suction filtration. Wash the crystals with a little cold solvent and dry between filter papers.

11.7 Determination of melting point

Melting points are ascertained in order to either check the purity of a sample of a compound or confirm the identity of the compound. The solid whose melting point it is required to determine is put on a white tile. A small thin-walled capilliary tube sealed at one end, known as a melting-point tube, is taken. The open end of the tube is pressed onto the solid, and the solid can be made to fall to the bottom of the tube by tapping the closed end of the tube on the tile. A column of solid 0.5 cm long is suitable.

The tube and contents are fixed as shown in diagram 11.7 by means of a rubber band. The boiling tube is slowly heated by means of a very low bunsen burner flame and the stirrer moved up and down to ensure even heating. Watch the crystals in the melting point tube carefully and the moment they melt, make a note of the temperature. Note also the temperature at which melting is complete. For pure substances this melting range is narrow.

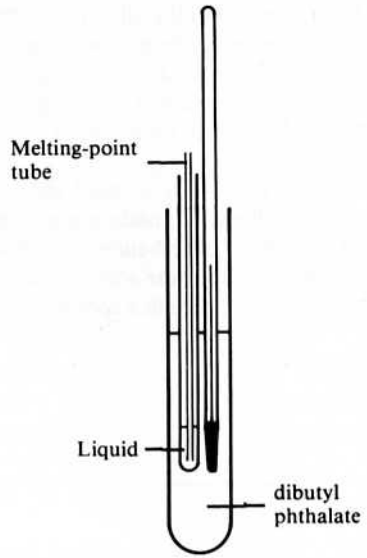


11.7

11.8 Determination of boiling point

Under normal conditions, the boiling point of a pure liquid has a constant value, so the boiling point can be used as a check on either purity or identity of a compound. If a reasonable volume of a liquid is available, the apparatus in diagram 11.1 can be used.

If only a small quantity of liquid is available, its boiling point may be determined by using the apparatus in 11.8. The open end of a melting point tube is immersed in a few drops of the liquid in a small thin-walled test-tube. The test-tube is fixed to the thermometer by an elastic band and heated slowly in the liquid bath. Near the boiling point, vapour escapes from the mouth of the melting point tube in a continuous stream of bubbles. At this point the apparatus is allowed to cool and the temperature at which the liquid rises into the melting point tube is taken as the boiling point.



11.8