

E24 PURIFICATION OF ORGANIC COMPOUNDS

Distillation, recrystallisation, melting and boiling point determination

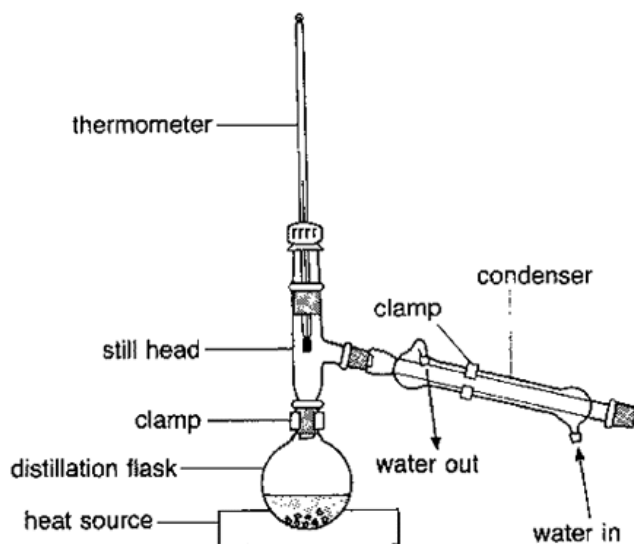
Each year, many thousands of new or unknown organic compounds are prepared or extracted from natural sources such as plants, fungi and animals. In order to identify an unknown organic compound or the components of a mixture of organic compounds, it is necessary firstly to separate and purify the compounds.

Purification of a liquid

At any given temperature, a certain number of molecules of a substance in the liquid state have sufficient energy to escape from the surface to create a vapour pressure. This tendency becomes greater as the kinetic energy of the molecules is increased (by raising the temperature). When a liquid is heated to a temperature at which the vapour pressure equals that of the surrounding atmosphere, the liquid boils. This temperature is known as the boiling point. The boiling point of a pure substance at a specified pressure is a characteristic property of that substance, and can be used as a criterion of purity and a means of identification.

Distillation is the process of vaporising a sample, condensing the vapour, and collecting the condensate in another vessel. Each volatile component of a mixture can be distilled from the reaction mixture in an increased state of purity by slowly increasing the temperature of the mixture and collecting each "fraction" that boils off. If the properties of the components of the mixture do not permit a clean separation, the products of the various fractions can be redistilled to improve their purity. In this way, mixtures of liquids with different vapour pressures at a given temperature can be separated or recovered from less volatile contaminants.

Summary - the basic principle involved in distillation is that often each of the component liquids in a mixture of liquids boils at a different temperature. A desired compound can be isolated from a mixture by slowly heating the mixture until the boiling point of that particular substance is reached, and collecting that fraction which boils off at this temperature. The condensate collected should contain only the desired compound and some small quantity of a higher boiling impurity. The diagram below shows the set up of a simple distillation apparatus.



Purification of a solid

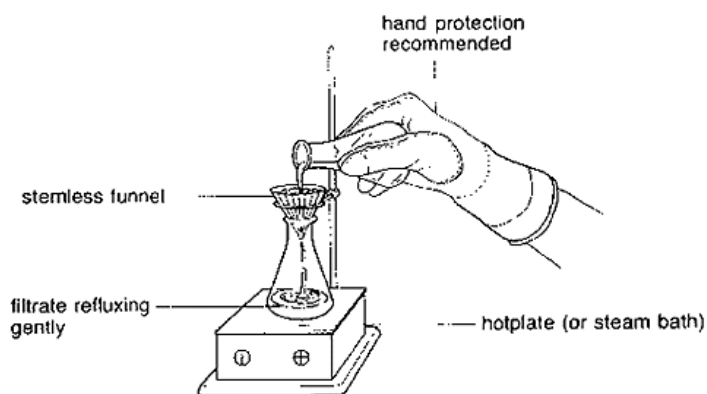
Separation of a mixture of solids or purification of solids is most readily achieved by **recrystallisation**. This process involves first identifying a suitable solvent in which the material to be recrystallised or purified has high solubility at elevated temperature (typically near the boiling point of the solvent) and low solubility at low temperature. The solid (or mixture) is dissolved in a minimal volume of hot solvent, filtered to remove any insoluble components in the mixture and the solution is slowly cooled to allow the product to crystallise.

The formation and growth of crystals is a complex process. It involves the removal of molecules from solution (molecule by molecule) and their stacking into a regular condensed state, which becomes a crystal lattice. Like molecules tend to pack more efficiently so the growth of crystals tends to bring together molecules of the same type. The effect of dissolving a solid and then permitting it to crystallise is that the crystals which form contain less impurities than the material which was originally dissolved. Successive recrystallisations can produce extremely pure crystalline samples.

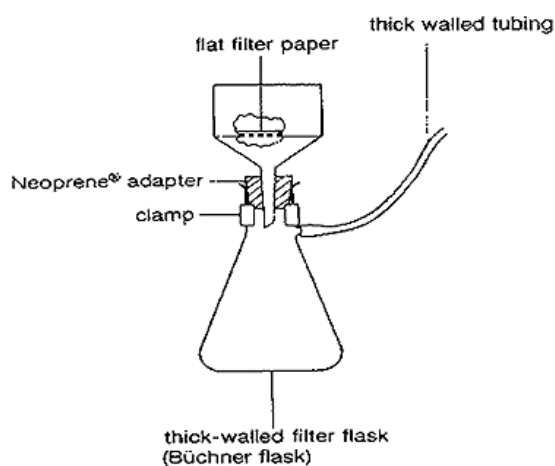
Recrystallisation is the purification method of choice where the solid is highly crystalline and forms crystals readily. It may also be carried out in industrial chemistry on a massive scale, e.g. the purification of white sugar from raw sugar.

Summary - the basic principle employed in recrystallisation is the dissolution of the crude material in a suitable hot solvent, filtration (1) while hot to remove any insoluble impurities, then cooling of the solution so that the required compound preferentially crystallises, leaving the soluble impurities in the solution (known as the "mother liquor"). These pure crystals can then be filtered off (2). The diagrams below show the two filtration processes (1) and (2).

A flow chart of this process is shown in Appendix 1.

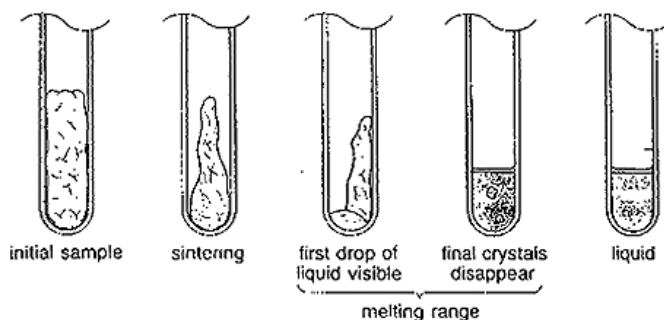


Filtration Process (1)



Filtration Process (2)

As in the case with liquids, a solid melts at a particular temperature. Unlike liquids, however, a recrystallised solid may still contain impurities in the form of solvent molecules trapped within the crystal lattice. Consequently, the melting point of a solid can be used as a measure of purity but not as a means of separation. Another effect of these impurities is that solids rarely melt at one temperature but instead melt over a range of temperatures. The typical changes that occur to a solid as it melts are shown below.



APPENDIX 1

Recrystallisation

The process of recrystallisation is set out below as a flowchart for easier assimilation.

