Separation Techniques

❖Most materials in our surroundings are mixtures of two or more components.

❖Mixtures are either homogeneous or heterogeneous. Homogeneous mixtures are uniform in composition, but heterogeneous mixtures are not.

❖Homogeneous and heterogeneous mixtures can be separated into their components by several physical methods.

❖The choice of separation techniques is based on the type of mixture and difference in the chemical properties of the constituents of a mixture.

Separation Techniques

❖Various types of separation processes are:

- Crystallization
- Precipitation
- Filtration
- Decantation
- Centrifugation
- Sublimation
- Evaporation
- Simple distillation
- Fractional distillation
- Chromatography
- Extraction
- Magnetic separation

❖Crystallization is the process of formation of solid crystals precipitating from a solution.

❖Crystallization is a technique which chemists use to purify solid compounds.

❖It is one of the fundamental procedures each chemist must master to become proficient in the laboratory.

❖Crystallization is based on the principles of solubility: compounds (solutes) tend to be more soluble in hot liquids (solvents) than they are in cold liquids.

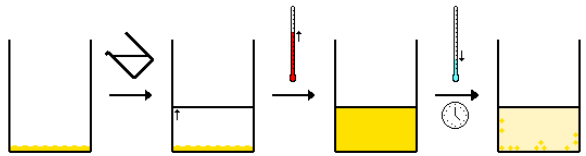
❖If a saturated hot solution is allowed to cool, the solute is no longer soluble in the solvent and forms crystals of pure compound.

❖Impurities are excluded from the growing crystals and the pure solid crystals can be separated from the dissolved impurities by filtration.

❖Recrystallization is a procedure for purifying compounds.

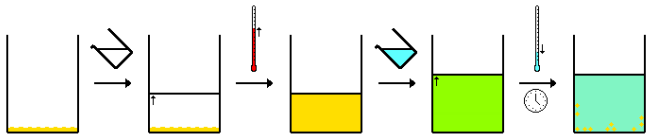
❖The most typical situation is that a desired "compound A" is contaminated by a small amount of "impurity B".

❖Single-solvent recrystallization; Typically, the mixture of "compound A" and "impurity B" are dissolved in the smallest amount of hot solvent to fully dissolve the mixture, thus making a saturated solution. The solution is then allowed to cool. As the solution cools the solubility of compounds in solution drops. This results in the desired compound recrystallizing from solution. The slower the rate of cooling, the bigger the crystals form.



❖Multi-solvent recrystallization

❖This method is the same as single solvent recrystallization but where two (or more) solvents are used. This relies on both "compound A" and "impurity B" being soluble in a first solvent. A second solvent is slowly added. Either "compound A" or "impurity B" will be insoluble in this solvent and precipitate, whilst the other of "compound A"/"impurity B" will remain in solution. Thus the proportion of first and second solvents is critical. Typically the second solvent is added slowly until one of the compounds begins to crystallize from solution and then the solution is cooled. Heating is not required for this technique but can be used.



❖Crystallization requires an initiation step.

❖This can be spontaneous or can be done by adding a small amount of the pure compound (a seed crystal) to the saturated solution, or can be done by simply scratching the glass surface to create a seeding surface for crystal growth.

❖Precipitated solids usually removed by using filtration.

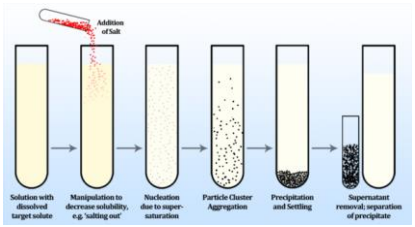
❖Filtration is the process of separating suspended solid matter from a liquid, by causing the latter to pass through the pores of some substance, called a filter.

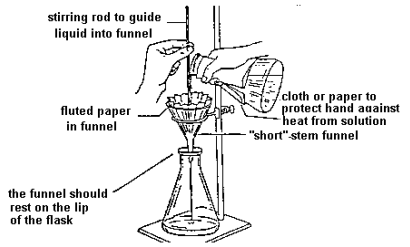
❖ The liquid which has passed through the filter is called the filtrate. The filter may be paper, cloth, cotton-wool, asbestos, slag- or glass-wool, unglazed earthenware, sand, or other porous material.

❖Precipitation is the formation of a solid in a solution or inside another solid during a chemical reaction or by diffusion in a solid.

❖When the reaction occurs in a liquid solution, the solid formed is called the precipitate. The chemical that causes the solid to form is called the precipitant.

❖Precipitation reactions can be used for making pigments, removing salts from water in water treatment, and in classical qualitative inorganic analysis.





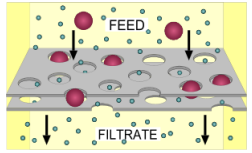
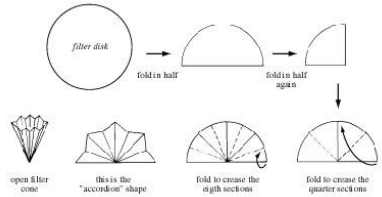
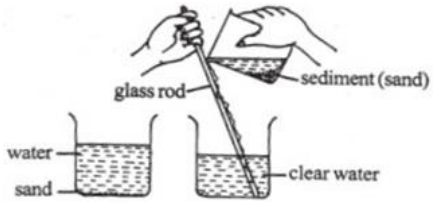


Figure 1.1 Rapid filtration of a hot solution using fluted filter paper



❖Decantation is a process for the separation of mixtures, by removing a top layer of liquid from which a precipitate has settled.

❖Usually a small amount of solution must be left in the container, and care must be taken to prevent a small amount of precipitate from flowing with the solution out of the container.



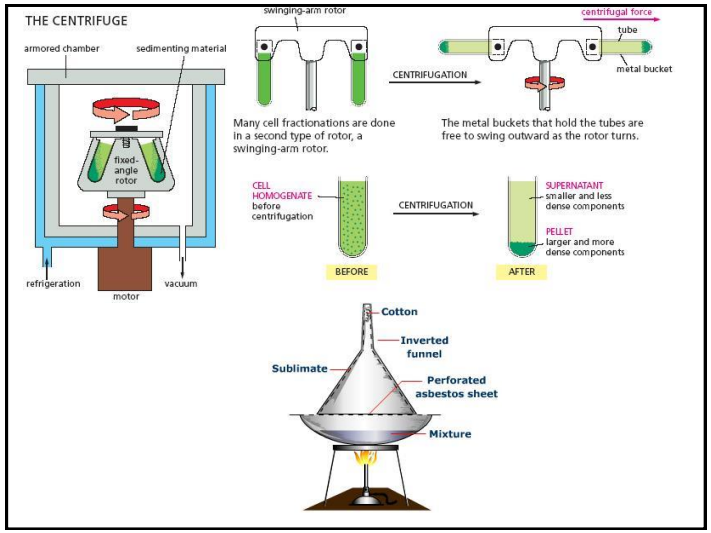
❖Centrifugation is a process that involves the use of the centrifugal force for the sedimentation of mixtures with a centrifuge.

❖More-dense components of the mixture migrate away from the axis of the centrifuge, while less-dense components of the mixture migrate towards the axis.

❖Chemists and biologists may increase the effective gravitational force on a test tube so as to more rapidly and completely cause the precipitate ("pellet") to gather on the bottom of the tube.

❖The remaining solution is properly called the "supernate" or "supernatant liquid".

❖The supernatant liquid is then either quickly decanted from the tube without disturbing the precipitate, or withdrawn with a Pasteur pipette.



❖Sublimation is the change of a solid substance directly to a vapor without first passing through the liquid state. The term is also used to describe the reverse process of the gas changing directly to the solid again upon cooling.

❖An example of sublimation is seen when iodine, on being heated, changes from a dark solid to a purplish vapor that condenses directly to a crystalline solid upon striking a cool surface. In this way pure crystals of iodine are prepared.

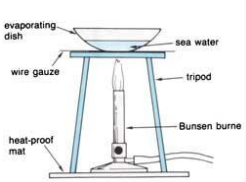
❖Some other substances, e.g., mercuric chloride, can be prepared by sublimation. Solid carbon dioxide, commonly known as dry ice, sublimes at - 78.5°C

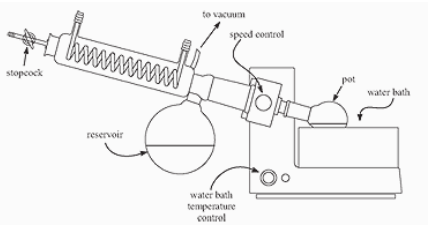
❖Sublimation also occurs when air saturated with water vapor is suddenly cooled below the freezing point of water. Frost and snowflakes are thus formed by water changing directly from the gaseous to the solid state.

❖Evaporation is a thermal separation process, widely used for concentration of liquids in the form of solutions, suspensions, and emulsions.

❖Concentration is accomplished by boiling out a solvent, normally water, from the liquid.

❖In most cases, concentrate resulting from the evaporation process is the final product. Sometimes, however, the evaporated, volatile component is also a main product, as, for example, during solvent recovery.





❖Simple distillation is a method used for the separation of components of a mixture containing two miscible liquids that boil without decomposition and have sufficient difference in their boiling points.

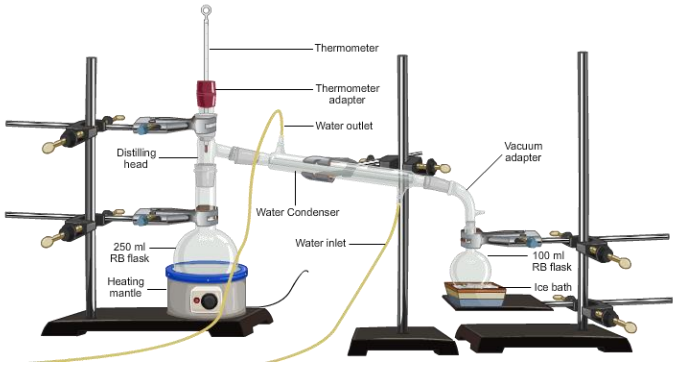
❖The distillation process involves heating a liquid to its boiling points, and transferring the vapors into the cold portion of the apparatus, then condensing the vapors and collecting the condensed liquid in a container.

❖In this process, when the temperature of a liquid rises, the vapor pressure of the liquid increases.

❖When the vapor pressure of the liquid and the atmospheric pressure reach the same level, the liquid passes into its vapor state.

❖The vapors pass over the heated portion of the apparatus until they come into contact with the cold surface of the water-cooled condenser.

❖ When the vapor cools, it condenses and passes down the condenser and is collected into a receiver through the vacuum adapter.



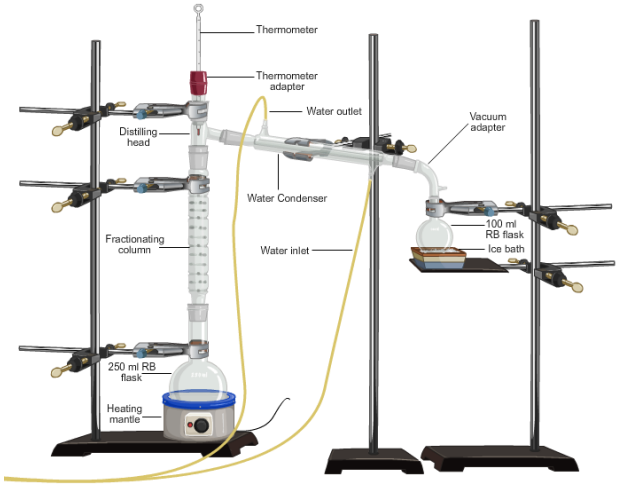
❖Fractional distillation: Fractional distillation is used for the separation of a mixture of two or more miscible liquids for which the difference in boiling points is less than 25K.

❖The apparatus for fractional distillation is similar to that of simple distillation, except that a fractionating column is fitted in between the distillation flask and the condenser.

❖A simple fractionating column is a tube packed with glass beads. The beads provide surface for the vapors to cool and condense repeatedly.

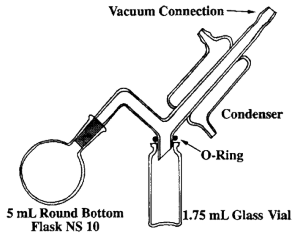
❖When vapors of a mixture are passed through the fractionating column, because of the repeated condensation and evaporation, the vapors of the liquid with the lower boiling point first pass out of the fractionating column, condense and are collected in the receiver flask.

❖The other liquid, with a slightly higher boiling point, can be collected in similar fashion in another receiver flask.



❖Vacuum distillation is a method of distillation whereby the pressure above the liquid mixture to be distilled is reduced to less than its vapor pressure (usually less than atmospheric pressure) causing evaporation of the most volatile liquid(s) (those with the lowest boiling points).

❖This distillation method works on the principle that boiling occurs when the vapor pressure of a liquid exceeds the ambient pressure. Vacuum distillation is used with or without heating the mixture.

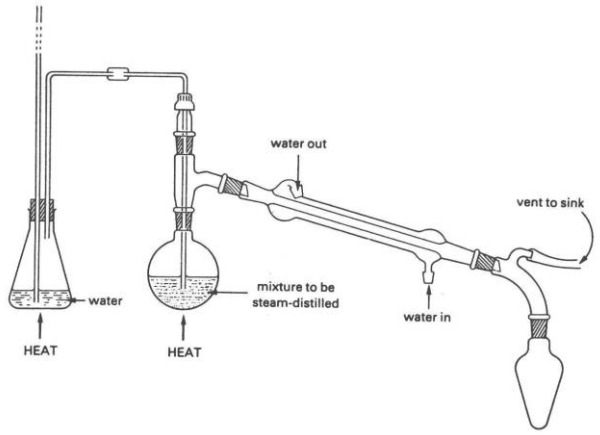


❖Like vacuum distillation, steam distillation is a method for distilling compounds which are heat-sensitive.

❖The temperature of the steam is easier to control than the surface of a heating element, and allows a high rate of heat transfer without heating at a very high temperature.

❖This process involves bubbling steam through a heated mixture of the raw material. By Raoult's law, some of the target compound will vaporize (in accordance with its partial pressure). The vapor mixture is cooled and condensed, usually yielding a layer of oil and a layer of water.

❖Steam distillation of various aromatic herbs and flowers can result in two products; an essential oil as well as a watery herbal distillate. The essential oils are often used in perfumery and aromatherapy while the watery distillates have many applications in aromatherapy, food processing and skin care.



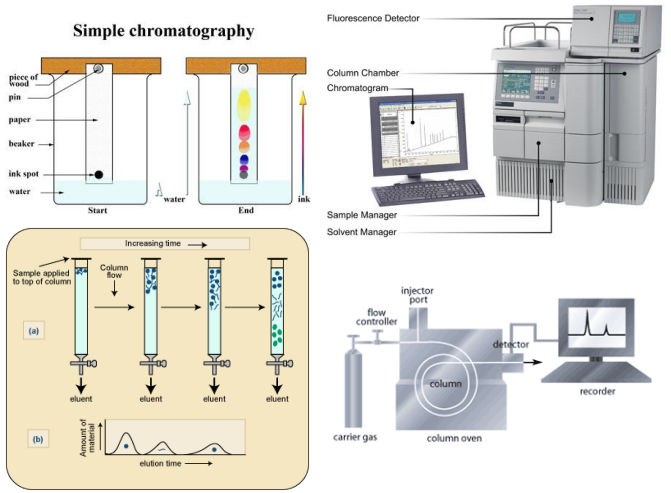
❖Chromatography is a separation technique used to separate the different components in a liquid mixture.

❖Chromatography involves the sample being dissolved in a particular solvent called mobile phase. The mobile phase may be a gas or liquid.

❖The mobile phase is then passed through another phase called stationary phase. The stationary phase may be a solid packed in a glass plate or a piece of chromatography paper.

❖The various components of the mixture travel at different speeds, causing them to separate.

❖There are different types of chromatographic techniques such as column chromatography, TLC, paper chromatography, and gas chromatography.



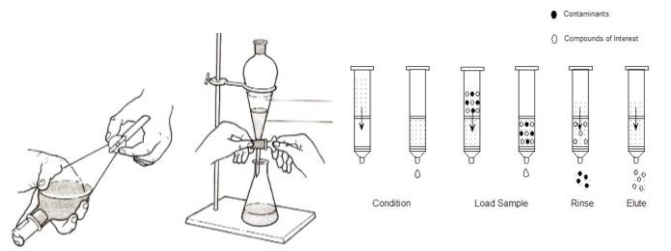
❖Extraction in chemistry is a separation process consisting in the separation of a substance from a matrix. Extractions use two immiscible phases to separate a solute from one phase into the other.

❖The distribution of a solute between two phases is an equilibrium condition described by partition theory.

❖Typical lab extractions are of organic compounds out of an aqueous phase and into an organic phase.

❖Liquid–liquid extraction also known as solvent extraction and partitioning, is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent.

❖It is an extraction of a substance from one liquid into another liquid phase. Liquid–liquid extraction is a basic technique in chemical laboratories, where it is performed using a separatory funnel.



❖Solid phase extraction is a chromatographic technique used to prepare samples for subsequent analysis by removing interfering substances that may be present.

❖This is done either by retaining the substance of interest and washing off everything else or by retaining the interfering substances and eluting the product of interest.

❖Magnetic separation is a process in which magnetically susceptible material is extracted from a mixture using a magnetic force.

❖This separation technique can be useful in mining iron as it is attracted to a magnet.

❖Magnetic beads are used for separation of various materials in biotechnology.