Chapter 2

Experimental Techniques in Chemistry

Filtration

Definition

The process of filtration is used to separate insoluble particles from liquids.

Filter Media

The filter media used are:

a. Filter paper

b. Filter crucible

Nature of the precipitate and other factors dictate which filter medium must be used.

Filtration through Filter Paper

Filtration by a glass funnel and filter paper is usually a slow process. Filter papers are available in a variety of porosites (pore sizes). Which pore size is to be used, depends upon the size of particles in the precipitate.

Procedure

1. The mixture is poured onto the filter paper.
2. The solvent (water) passes through leaving behind the suspended particles on the filter paper. 3. The filter paper should be large enough so that it is one-fourth to one-half full of precipitate at the end of filtration.
4. The funnel should be large enough for its rim to extend 1 to 2 cm above the top circumference of the paper.
5. The stem of the funnel should remain continuously full of liquid as long as there is liquid in the conical portion.
6. The stem of the funnel should be several inches long so that it can extend a few centimeters down into the receiving beaker.
7. The tip should touch the side of the beaker. The filtrate runs down the side of beaker without splashing.

Folding of Filter Paper

The paper should be folded twice.

The first fold should be along the diameter of the paper.

The second fold should be such that edges do not quite match.

The paper should be opened on the slightly larger section. This provides a cone with three fold thickness halfway around and one thickness the other halfway around.

An apex angle very slightly greater than 60 degrees is formed.

The paper is inserted into 60 degree funnel, moistened with water and firmly pressed down.

The filtering operation should be aided by a gentle suction as liquid passes through the stem. This suction cannot develop unless the paper tightly fits all around its upper circumference.

Fluted Filter Paper

Ordinary filter paper is folded in such a way that a fan like arrangement with alternate elevations and depressions at various folds is obtained.

Advantage

The rate of filtration through conical funnel can be considerably increased using a fluted filter paper.

Filtration through Filter Crucibles

Another convenient way to filter a precipitate is by suction through a crucible.

Gooch Crucible

Assembly

It is made of porcelain having a perforated bottom which is covered with paper pulp or a filter paper cut to its size. Quick filtration can be done by placing the Gooch crucible in a suction filtering apparatus.

Advantage

1. It is useful for the filtration of precipitates, which need to be ignited at high temperature.
2. Concentrated HCl and $KMnO_4$ solutions are filtered by covering its perforations with asbestos mat.

Sintered Glass Crucible

Assembly

Sintered glass crucible is a glass crucible with a porous glass disc sealed into the bottom.

Advantage

It is very convenient to use because no preparation is needed as with the gooch crucible.

Crystallization

Definition

Crystallization is the removal of a solid from solution by increasing its concentration above the saturation point in such a manner that the excess solid separates out in the form of crystals.

Principle

The solute should be soluble in a suitable solvent at high temperature and the excess amount of the solute is thrown out as crystals when it is cooled.

Choice of a Solvent

Following are the characteristics of an ideal solvent:

i. It should dissolve a large amount of the substance at its boiling point and only a small amount at the room temperature.
ii. It should not react chemically with the solute.
iii. It should either not dissolve the impurities or the impurities should not crystallize from it along with the solute.
iv. On cooling it should deposit well-formed crystals of the pure compound.
v. It should be inexpensive.
vi. It should be safe to use and should be easily removable.

Solvents Commonly Employed

Water, rectified spirit (95% ethanol), absolute ethanol, diethyl ether, acetone, chloroform, carbon tetrachloride, acetic acid and petroleum ether.

Preparation of the Saturated Solution

The substance is dissolved in a minimum amount of a suitable solvent and is heated directly or on a water bath with constant stirring. More solvent is added to the boiling solution until all the solute has dissolved.

Filtration

The hot saturated solution is filtered through a normal or a fluted filter paper to avoid the premature crystallization of the solute on the filter paper or in the funnel stem. Hot water funnel is used for this purpose.

Cooling

The hot filtered solution is then cooled at a moderate rate so that medium sized crystals are formed. Slow cooling yields bigger crystals which include solvent and impurities.

Collecting the Crystals

When the crystallization is complete, the mixture of crystals and the mother liquor is filtered through a Gooch crucible using a vacuum pump with full suction. The filter cake is pressed firmly with a cork to drain the left-

over liquid. The crystals are washed with a small portion of cold solvent and the process is repeated several times. The mother liquor is concentrated by evaporation and cooled to obtain a fresh crop of crystals.

Drying of the Crystallized Substance

The drying methods employed are:

1. Pressing it between several folds of filter papers and repeating the process several times.

Disadvantage

The crystals are crushed to a fine powder and sometimes the fibres of filter paper contaminate the product.

2. The crystals are dried in an oven provided the substance does not melt or decompose on heating at 100° C.

Best Method

3. A safe and reliable method of drying crystals is through a vacuum desiccator. The crystals are spread over a watch glass and kept in a vacuum desiccator for several hours.

Drying agents

$CaCl_2$, silica gel or phosphorus pentaoxide.

Decolourization of Undesirable Colours

The colouring matter or resinous products make the product coloured on crystallization. The product is boiled in the solvent with finely powdered animal charcoal. The hot solution is then filtered. The coloured impurities are adsorbed by animal charcoal and the pure decolourized substance crystallizes out on cooling.

Sublimation

Definition

It is a process in which a solid, when heated, vapourizes directly without passing through the liquid phase and these vapours can be condensed to form the solid again.

Use

It is frequently used to purify a solid.

Examples

Ammonium chloride, iodine, naphthalene, benzoic acid.

Procedure

The substance is taken in a watch glass covered with an inverted funnel. The substance is then heated slowly over a sand-bath and the funnel is cooled with wet cotton. The pure solid deposits on the inner side of the funnel.

Solvent Extraction

Definition

A solute can be separated from a solution by shaking the solution with a solvent in which the solute is more soluble and the added solvent does not mix with the solution.

Apparatus used

Usually it is done by placing the solution and the second liquid into a separating funnel.

Example

Ether extraction

This is used to separate the products of organic synthesis from water. The aqueous solution containing the organic product is shaken up with ether in a separating funnel and allowed to separate.

Aqueous phase: Inorganic impurities
Ether layer by evaporation: Organic product

Efficiency of the process

Repeated extractions using small portions of solvent are more efficient than using a single but larger volume of solvent.

Applicability

The technique is particularly useful when the product is volatile or thermally unstable.

Distribution law or Partition law

This law states that a solute distributes itself between two immiscible liquids in a constant ratio of concentrations irrespective of the amount of solute added.

Example

The distribution of iodine between two immiscible solvents, water in the presence of KI and carbon tetrachloride. Iodine reacts with iodide ion to produce tri-iodide ion in a reversible reaction.

$$I_2 + I^-(aq) \rightleftharpoons I_3^-(aq)$$

Soluble in CCl_4 Soluble in water

If we add CCl_4 to an aqueous solution of I_3^- ions, the iodine will transfer from the aqueous layer into the organic layer. The brown colour of the tri-iodide ions fades and the purple colour of free iodine molecules appears in organic phase. No matter how much iodine is used, the ratio of the final concentrations at equilibrium is constant. The constant is called distribution coefficient, K and is given by

$$K = \frac{[I_2(CCl_4)]}{[I_3^-(aq)]}$$

Chromatography

Definition

The word chromatography originates from the Greek word "Khromatos" meaning colour writing.

Primary use

Chromatography is a method used primarily for the separation of a sample of mixture.

It involves the distribution of a solute between a stationary phase and a mobile phase.

Stationary phase

The stationary phase may be a solid or a liquid supported as a thin film on the surface of an inert solid.

Mobile phase

The mobile phase flowing over the surface of the stationary phase may be a gas or a liquid.

In chromatography, substances are separated due to their relative affinities for the stationary and mobile phases.

Distribution coefficient

The distribution of the components of a mixture between the two phases is governed by distribution coefficient K.

$$K = \frac{\text{Concentration of a component in the moving phase}}{\text{Concentration of that component in the stationary phase}}$$

The component of a mixture with a small value of K mostly remains in the stationary phase. The component with a greater value of K remains largely dissolved in the mobile phase.

Types of chromatography

Adsorption chromatography

In it the stationary phase is a solid. A substance leaves the mobile phase to become adsorbed on the surface of the solid phase.

Partition chromatography

In it the stationary phase is a liquid. The substances being separated are distributed throughout both the stationary and mobile phases.

Techniques of Chromatography

Paper Chromatography

It is a technique of partition chromatography.

Stationary phase

The stationary phase is a liquid (say H_2O) adsorbed on paper. The adsorbed water behaves as an immiscible liquid towards the mobile phase, which passes over the paper.

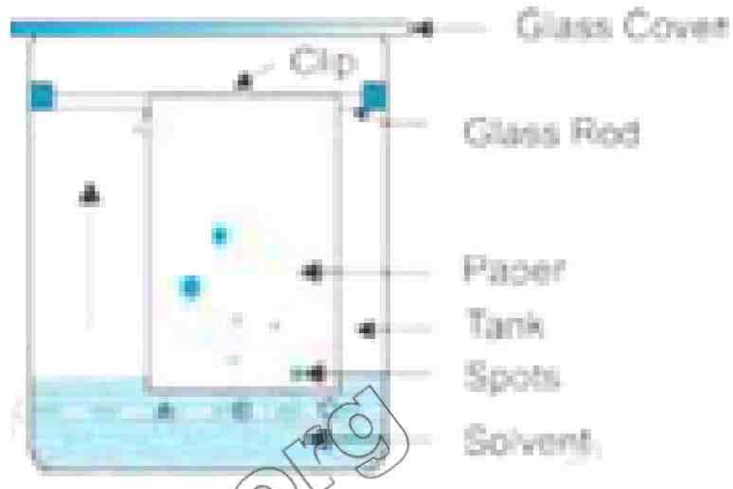
Mobile phase

The mobile phase is usually an organic liquid.

Methods to Perform Chromatography

(i) ascending (ii) descending (iii) radial/circular.

Procedure



1. The solvent is in a pool at the bottom of a vessel in which the paper is supported and the solvent travels upwards by capillary action.
2. A solvent mixture is poured into the chromatographic tank.
3. Cover the tank to homogenize its inner atmosphere.
4. Take about 20 cm strip of Whatmann's chromatographic paper No.1 and draw on it a thin pencil line about 2.5 cm from one end.
5. Spot a point, on the pencil line, with the sample mixture solution.
6. Spots of the known compounds may also be placed alongside for identification.
7. When the spots have dried, suspend the paper with clips so that the impregnated end dips into solvent mixture to a depth of 5-6 mm.
8. Cover the tank.
9. As the solvent front passes the spots, the solutes begin to move upward.
10. The rate at which they move depends on their distribution coefficients.
11. When the solvent front has risen to about $3/4^{th}$ of the length of the paper, remove the strip, mark the solvent front with a pencil and allow the strip to dry.
12. Once the paper is dried, the pattern on the paper is called a chromatogram.
13. The different components of the mixture, if coloured, can visually be identified.
14. If colourless, the chromatogram has to be developed by chemical methods or physical techniques used to identify the spots.
15. Each component has a specific retardation factor called R_f value. The R_f value is related to its distribution coefficient and is given by:

$$R_f = \frac{\text{Distance travelled by a component from the original spot}}{\text{Distance travelled by solvent from the original spot}}$$

Uses of Chromatography

- In organic synthesis for separation, isolation and purification of the products.
- In qualitative and quantitative analyses.
- For determination of the purity of a substance.