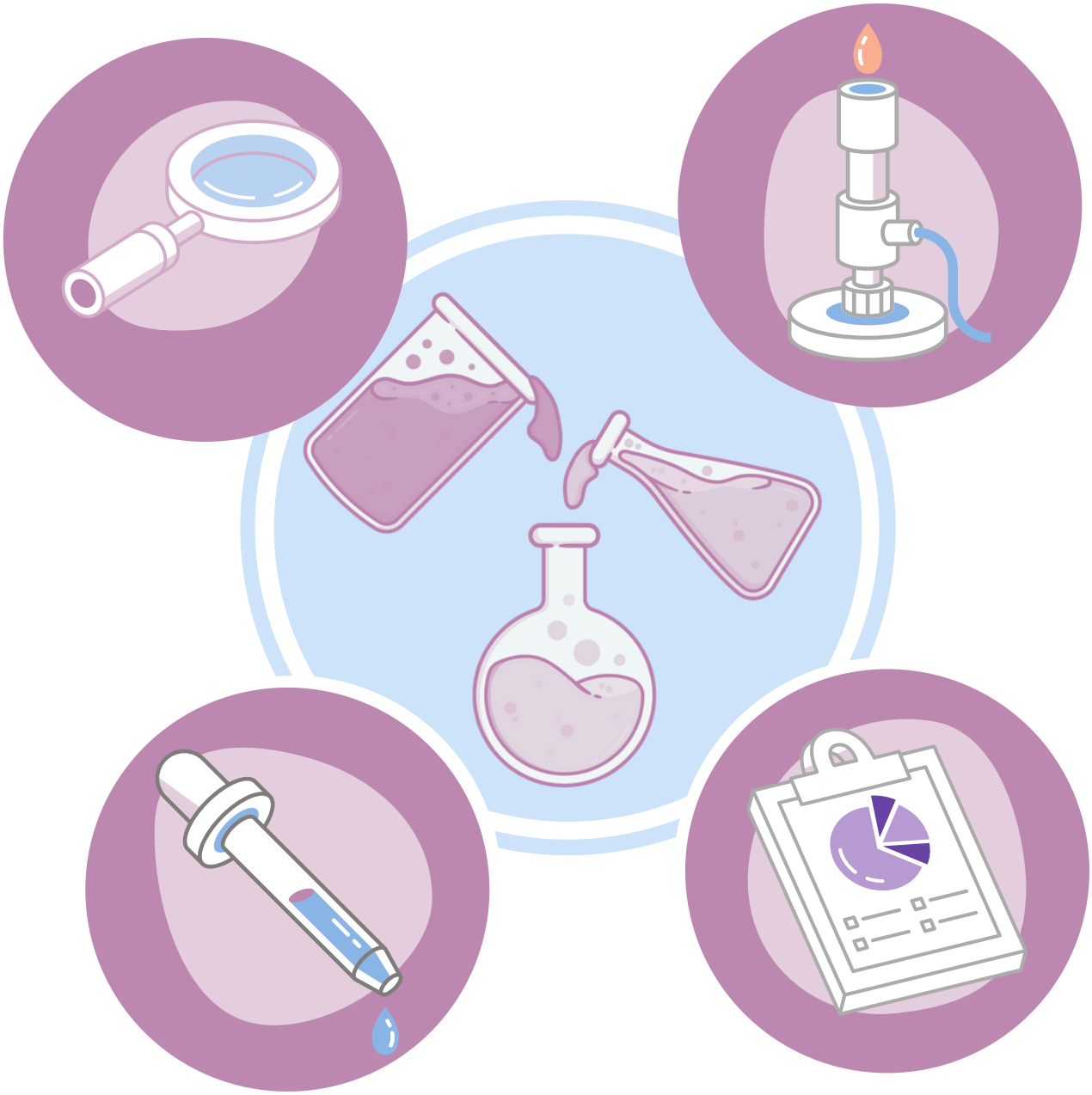
PreMed.PK

EXPERIMENTAL TECHNIQUES



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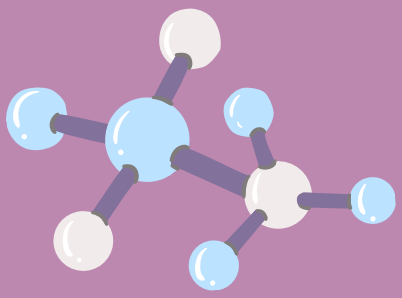


NOTES
EXPERIMENTAL TECHNIQUES IN CHEMISTRY

INTRODUCTION TO ANALYTICAL CHEMISTRY

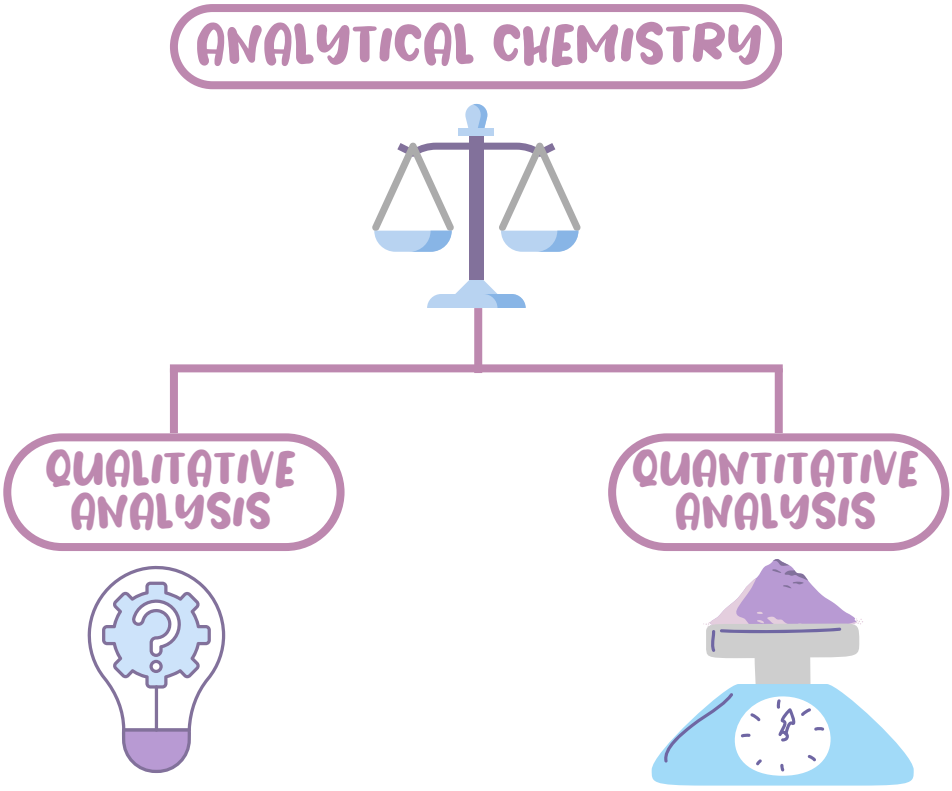
“Analytic chemistry is the science of chemical characterization”

It consist of:

1. Quantitative analysis- relative amount of element is determined
2. Qualitative analysis- concerned with detection/identification of substance

There are 4 steps for quantitative determination:

1. Obtaining a sample for analysis
2. Separation of desired constituent
3. Measurement and calculation of result
4. Drawing conclusion from the analysis

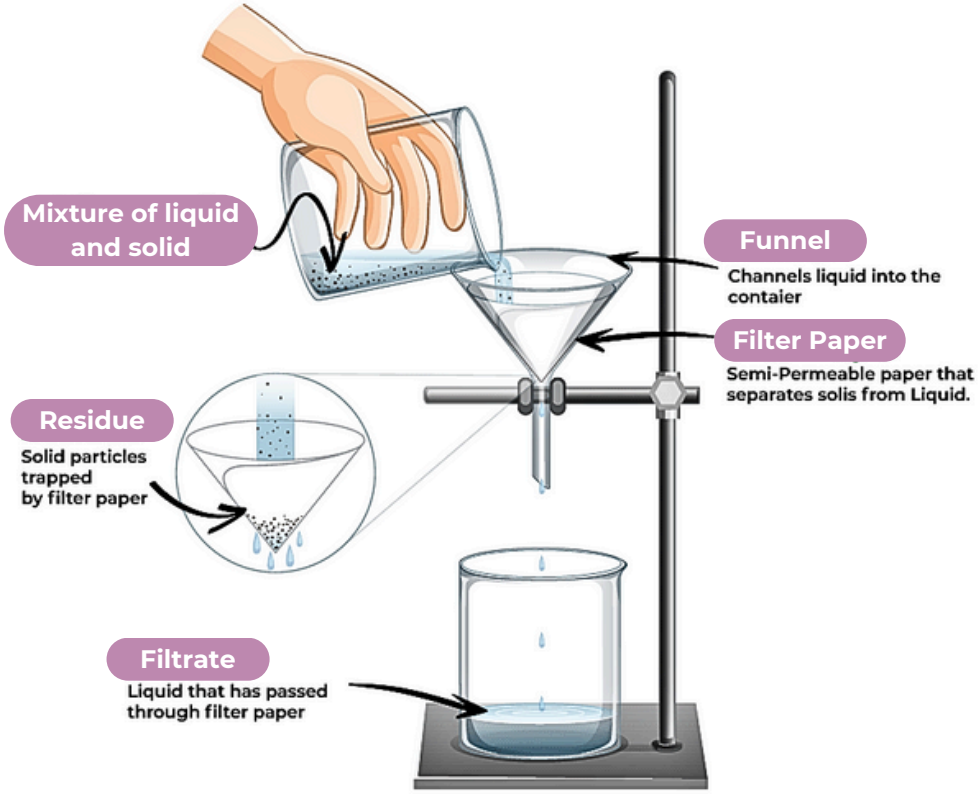


NOTES

FILTRATION

" Process of separation of insoluble particles from liquids."

It can be performed with several types of filter media. Nature of the precipitate and other factors dictate which filter medium must be used. There are many ways to do so but the most convenient way is either through filter paper or filter crucible.



? TEST YOURSELF

Q.1 FLUTED FILTER PAPER IS USED TO _____

a) Filter hot solution
b) Avoid premature crystallization
c) Increases rate of filtration
d) Decrease the area

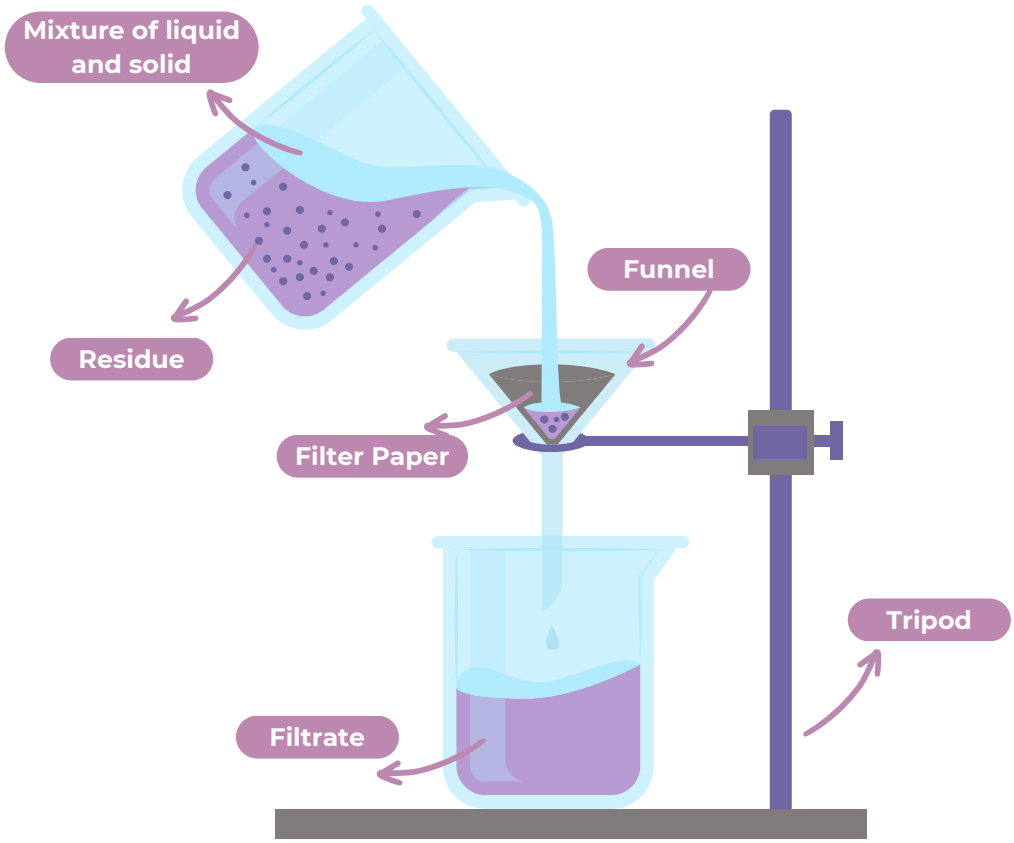
NOTES

Filtration through Filter Paper:

It is usually a slow process

Procedure:

- When the mixture is poured onto the filter paper, the solvent passes leaving behind the suspended particles on the filter paper.
- Filter paper are available in variety of pore sizes and the pore size which is to be used depends upon the size of precipitate.
- The filter paper should be large enough so it fits 1/4th to one-half full of precipitate at the end of filtration.
- The funnel should be large enough for its rim to extend 1 to 2 cm above the circumference of the paper.
- If the process of filtration is to run smoothly, the stem of funnel should remain continuously full of liquid as long as there is liquid in bathe conical portion.
- The stem of the funnel should be several inches long so that it can extend a few centimeters down into the receiving beaker, and the tip should touch the side of beaker.
- In this way, filtrate runs down the beaker without splashing.



NOTES

Folding of Filter Paper:

- The paper should be folded twice.
- The 1st fold should be along the diameter of the paper.
- The second fold should be such that edges don't quite match.
- The paper should be open on a slightly larger section. This provides a cone with three fold thickness halfway around and one thickness the other halfway around and apex angle very slightly greater than 60 degree.
- The paper must be inserted into 60 degree funnel, moistened with water and firmly pressed down.
- Gentle suction during this process makes it less time consuming. This suction cannot develop unless the paper fits tightly around its upper circumference.
- The paper should be folded twice.
- The 1st fold should be along the diameter of the paper.
- The second fold should be such that edges don't quite match.
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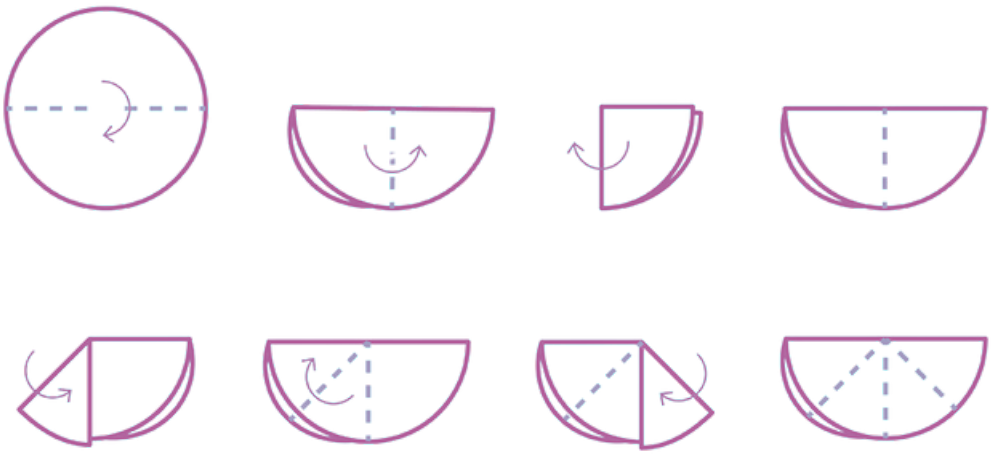
- The paper must be inserted into 60 degree funnel, moistened with water and firmly pressed down.
- Gentle suction during this process makes it less time consuming. This suction cannot develop unless the paper fits tightly around its upper circumference.

Fluted filter paper:

This increases the rate of filtration through conical funnel.

Procedure:

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



Folding of Filter Paper

NOTES

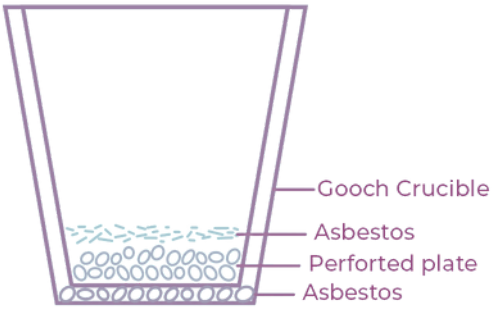
Filtration through Filter crucible:

1.Gooch crucible:

- **Apparatus:**

It is made up of porcelain having a perforated bottom which is covered with paper pulp or filter paper cut to its size.

- **Process:**
- Quick filtration can be done by placing the Gooch crucible in a suction filtering apparatus.
- If it's perforation are covered with asbestos mat, then it may be used to filter conc. HCl and KMNO4 solutions.

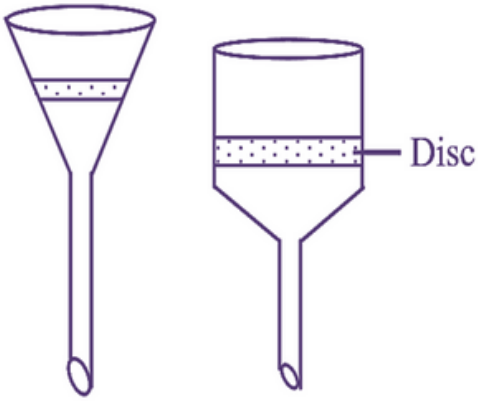


Gooche Crucible

2.Sintered glass crucible:

It is a glass crucible with a porous glass disc sealed at bottom.

- **Use:**
- convenient to use because no preparation is needed for it.



Sintered glass Crucible

TEST YOURSELF

Q.2 WHAT TYPE OF MIXTURE IS SEPARATED BY FILTRATION?___

a) Solid-liquid mixture
b) Liquid -liquid mixture
c) Solid-solid mixture
d) Solid- gas mixture

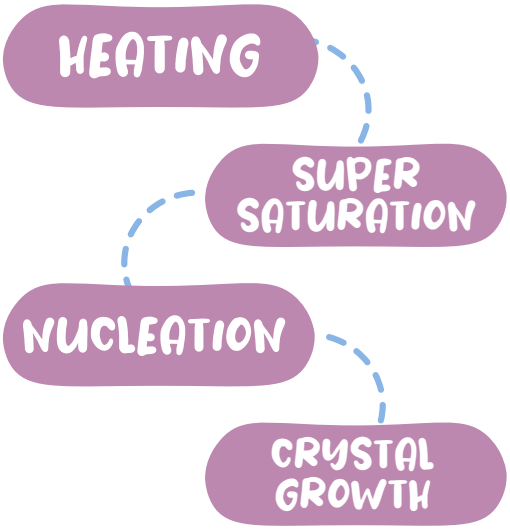
NOTES

CRYSTALLIZATION

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

Purpose:
Purification of a crude product which is made during the preparation of chemical compound.

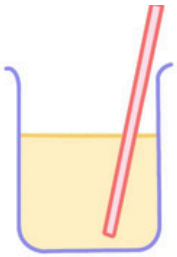
Principle:
The solute should be soluble in suitable solvent at high temperature and the excess amount of solute is thrown out as crystals when it is cooled.



Mechanism of Crystal Growth

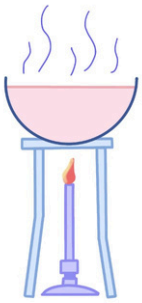
Crystallisation:

step 1



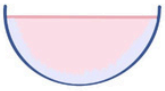
Stir to dissolve solid

step 2



The solution is heated to dissolve most of the solid

step 3



The solution is allowed to cool to obtain crystals

step 4

The cold solution is poured to obtain the crystals by pressing them between the sheets of filter paper

CRYSTALLIZATION

The steps for crystallization are following:

1. Choice of a Solvent:

Features of an ideal solution:

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

Solvents for crystallization:

- Water
- Rectified spirit (95% ethanol)
- Absolute ethanol
- Acetone
- Chloroform
- Diethyl ether

2. Preparation of Saturated Solution:

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

3. Filtration:

- Normal or fluted filter paper is used to filter the hot saturated solution containing insoluble impurities.
- This avoids premature crystallization of the solute on the filter paper or in the funnel stem.
- If necessary hot water should be used for this purpose .

4. Cooling:

- **Rate of cooling ∝ 1/ size of crystals**
- For medium sized crystals, hot filtered solution is cooled for moderate rate.
- For large sized crystals, hot filtered solution is cooled at slow rate which are likely to include considerable amount of solvent carrying impurities with it and complicating the drying process.

5. Collecting the crystals:

- The mixture of crystals and mother liquor is filtered through Gooch crucible by using vacuum pump after the crystallization is completed.
- In order to drain mother liquor from crystals, full suction is applied.
- When the filter cake is rigid enough, it is pressed firmly with a cork to drain the left-over liquid.

NOTES

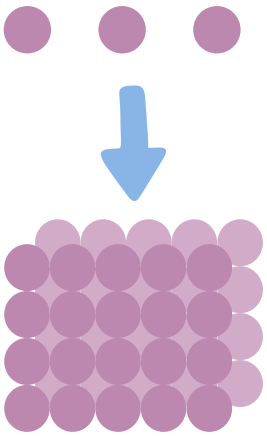
- The crystals are then washed with small portion of cooled solvent and the proceeds is repeated several times.
- The mother liquor is quite often concentrated by evaporation and cooled to obtain a fresh drop of crystals.
- **Success of this process ∝ Amount of crystallized product obtained from the crude product.**

6. Drying of the crystallized substance:

- Drying of crystallized substance is done by pressing the crystals between several folds of filter paper and repeating the process.
- Crystals when crushed to fine powder are sometimes contaminated by the fibers of filter paper.
- Alternatively, the crystals are dried in oven provided the substance does not melt or decompose on heating at 100 C.
- A safe and reliable method for drying crystals is through vacuum desiccator for several hours.
- The drying agents used in desiccator are CaCl2, silica gel or phosphorus pentoxide.

7. Decolorization of undesirable substances:

- The coloring matter or resinous products might affect the appearance the product and it may appeared colored during the preparation of crude product.
- These impurities are conveniently removed by boiling the substance in the solvent with the sufficient quantity of fined powdered animal charcoal and the filtering the hot solution.
- The colored impurities are adsorbed by the animal charcoals and the pure decolorized substance crystallized out on cooling.



TEST YOURSELF

Q.2 ONE OF THE MOST COMMON SOLVENTS USED FOR CRYSTALLIZATION IS

a) Water
b) Alcohol
c) Normal Saline
d) Sulphuric acid

NOTES

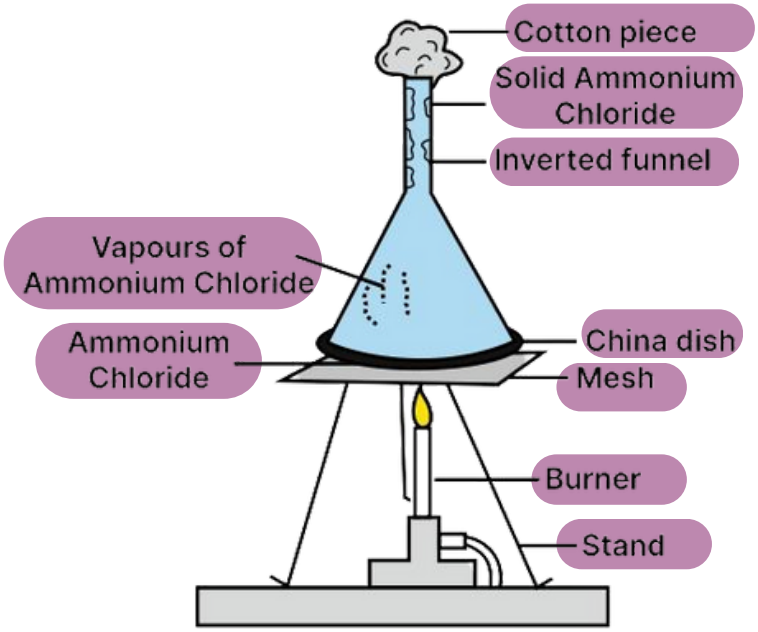
SUBLIMATION

"Conversion of a substance from the solid to gaseous state without its becoming liquid."

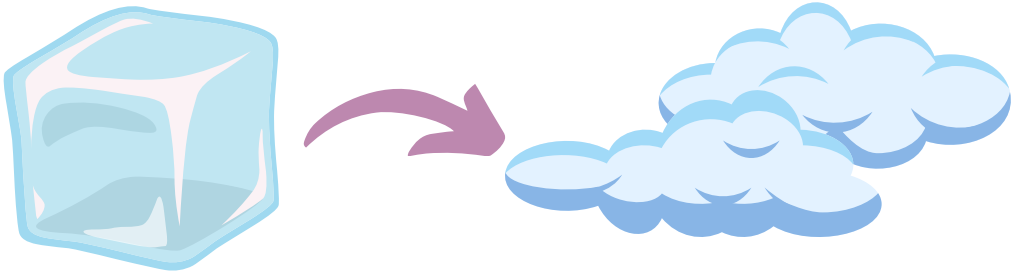
- It is frequently used to purify a solid.
- E.g: ammonium chloride, iodine, naphthalene, benzoic acid etc.

Procedure:

- The substance is taken in 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 inner side of funnel.



Process of Sublimation



Solid **Gas**

SOLVENT EXTRACTION

"Separation of a particular substance from a mixture by dissolving that substance in a solvent that will dissolve it, but which will not dissolve any other substance in the mixture."

- It is an equilibrium process and follows 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.
- Important technique in chemical analysis

Example:

Most common laboratory example of solvent extraction is of **ether extraction**.

- It is used to separate the products of organic synthesis from the water.
- The aqueous solution containing the organic product is shaken with ether in separating funnel and is allowed to separate.
- The inorganic impurities remains in aqueous phase where organic compound goes to ether layer.
- The ether layer is separated out and the organic compound is obtained by evaporating the ether.
- Repeating extraction using small solvent is more efficient than using single but large volume of solvent.
- This technique is useful when product is volatile or thermally unstable.

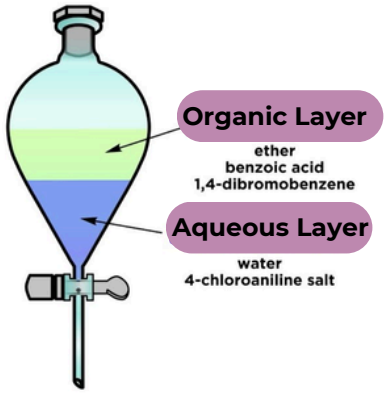
Example:

Consider, for example, the distribution of iodine between two immiscible solvents, water in the presence of KI and carbon tetrachloride. Iodine reacts with iodide ions to produce tri-iodide ions in a reversible reaction.
Rate at which iodine passes from CCl4 to water equals the rate at which it passes from water to CCl4 . So, if we add CCl4 to an aqueous solution of I 3 - ions, the iodine will transfer from the aqueous layer into the organic layer.
As a result,the brown color of the tri-iodide ions fades and the purple color of free iodine molecules appears in the organic phase.

To achieve a good separation, the two liquids are gently shaken to increase their area of contact and improve the chances of transferring iodine molecules. 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 :

$$K=[I_2(CCl_4)]/[I^-_{3(aq)}]$$



NOTES

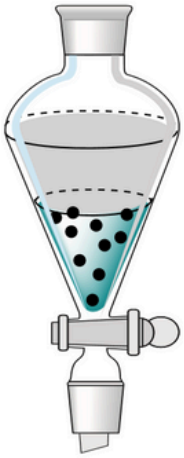
“ The method which is primarily used for the separation of sample of mixture.”

- The word chromatography originates from the greak word “Khromatos” meaning color writing
- It involves the distribution of a solute between stationary and mobile phase
- Substances are separated due to relative affinities for the stationary and mobile phase

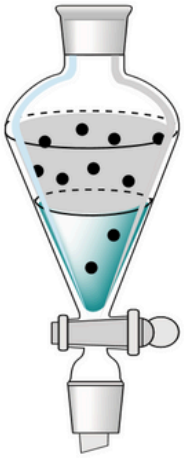
Stationary phase:

- It may be solid or liquid supported as thin film on the surface of inert solid.
- Mobile phase: It may be gas or a liquid flowing over the surface of stationary phase.
- The distribution of components of mixture between two phases is denoted by K: **K= Conc. of a component in the moving phase/Conc. of a component in the stationary phase**
- The component with small value of K remains mostly in stationary phase as moving phase flows over it.
- The component with greater value of K remains largely dissolved in mobile phase and quickly passes over stationary phase.

Add immiscible layer



Shake layers



CHROMATOGRAPHY

Types of Chromatography:

Adsorption chromatography:

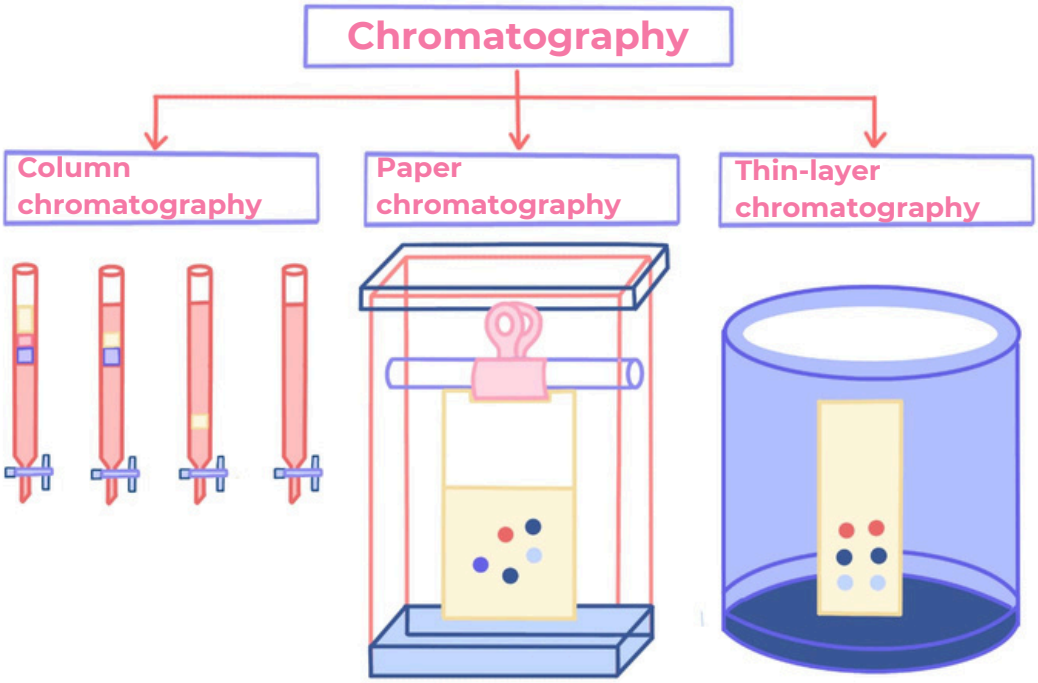
- Stationary phase is solid.
- Substance leaves the mobile phase to become adsorbed on the surface of solid phase.

Partition Chromatography:

- Stationary phase is liquid.
- Substances being separated are distributed throughout both stationary and mobile phases.

Paper Chromatography:

- One of the most common. technique in chromatography
- It is a technique of partition chromatography.
- Stationary phase is liquid adsorbed on the surface of water.
- Adsorbed water behaves as immiscible liquid towards the mobile phase which passes over the paper.
- The mobile phase is usually an organic liquid.



NOTES

Common ways to carry out paper chromatography:

1. Ascending
2. Descending
3. Radial/Circular

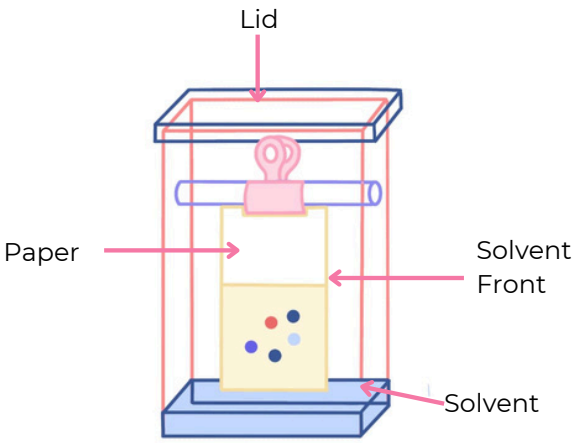
Ascending type involves the solvent being in pool at the bottom of a vessel in which paper is supported and the solvent travels upwards by capillary action.

Three main steps to perform it:
1) application of the sample,
2) "developing" the chromatogram by allowing the mobile phase to move up the paper.
3) calculating Rf values and making conclusions.
The distance traveled by the spot is measured to the MIDDLE of the spot.

Rf= Distance traveled by the component from the original spot/Distance traveled by the solvent from the original spot

Uses of Chromatography:

- Useful in separation, isolation and purification of the products.
- Important in both quantitative and qualitative analysis.
- Used for the determination of purity of a substance.



TEST YOURSELF

Q.2 THE PATTERN ON THE PAPER IN CHROMATOGRAPHY IS CALLED

a) Chroming
b) Chroma
c) Chromatograph
d) Chromatogram

ANSWER KEY

Q.1
Answer: c
Explanation: **Fluting the paper maximize the rate at which liquid may flow through the filter paper by increasing the surface area and allowing air to enter flask to permit pressure equalization.**

Q.2
Answer: a
Explanation: **Filtration is used for separating heterogeneous mixture. It is usually used for separating a solid-liquid mixture. It removes suspended solid matter from a liquid, by passing through the pores of filter. The liquid passed through the filter is termed as the filtrate.**

Q.3
Answer: a
Explanation: **Water is particularly common solvent to be found in crystals because it is small and polar. But all solvents can be found in some host crystals. Water is noteworthy because it is reactive, whereas other solvents such as benzene are considered to be chemically innocuous.**

Q.4
Answer: d
Explanation: **The correct answer is chromatogram**