Chapter 15

BASIC SEPARATION TECHNIQUES

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Student Learning Outcomes

After studying this chapter, students will be able to:

- Define important terms associated with creating chemical solutions: Solvent, Solute, Solution, Residue and Filtrate. **(Knowledge)**
- Explain methods of separation and purification: **(Understanding)**
  a) Filtration b) Crystallization
  c) Simple Distillation d) Fractional Distillation
- Identify substances and assess their purity using melting and boiling point information. **(Application)**
- Suggest suitable separation and purification techniques given information about the substances involved and their usage in daily life. **(Application)**
- Describe how paper chromatography is used to separate mixtures of soluble substances using suitable solvent. **(Understanding)**
- Describe use of locating agents when separating mixtures containing colorless substances. **(Understanding)**
- Interpret simple chromatograms to identify unknown substances by comparison with known substances pure and impure substances. **(Application)**
- State and use for R_f. **(Knowledge)**

➲ Analytical Chemistry

- **Analytical chemistry is the science of chemical characterization.**
- A complete chemical characterization of a compound must include both qualitative and quantitative analyses.

➢ Qualitative Analysis:

"In qualitative analysis, the chemist is concerned with the detection or identification of the elements present in a compound."

➢ Quantitative Analysis:

"In quantitative analysis, the relative amounts of elements in compounds are determined."

Differences between Qualitative and Quantitative Analysis

Qualitative Analysis	Quantitative Analysis
The detection or identification of different elements present in the compound is called as qualitative analysis.	The analysis in which the exact amount of each element present in the compound is determined is called as quantitative analysis.
It is the initial analysis to detect the elements present in the compound.	By this analysis, the % age of element present in the compound can be calculated.
Example: Salt analysis, detection of elements and functional groups.	**Example:** Volumetric analysis, combustion analysis, gravimetric analysis.

➢ Major Steps for Complete Chemical Characterization:

A complete quantitative determination generally consists of four major steps

(i) Obtaining a sample for analysis
(ii) Separation of the desired constituent
(iii) Measurement, and calculation of results
(iv) Drawing conclusion from the analysis.

Here, some basic separation techniques are discussed to practice in the lab.

METHODS OF SEPARATION OF MIXTURES

The components of a mixture can be separated by any of the following physical methods.

1. Filtration
2. Crystallization
3. Fractional distillation
4. Simple distillation
5. Paper Chromatography

➲ Filtration

- Filtration process is used to separate one of the components of a two-component heterogeneous mixture from a solution.
- ***"The process in which suspended solid particles or insoluble particles are separated from a liquid through a filter medium is called filtration."***
- The first step is to select a suitable solvent in which one of the components dissolves completely while the other remains practically insoluble.

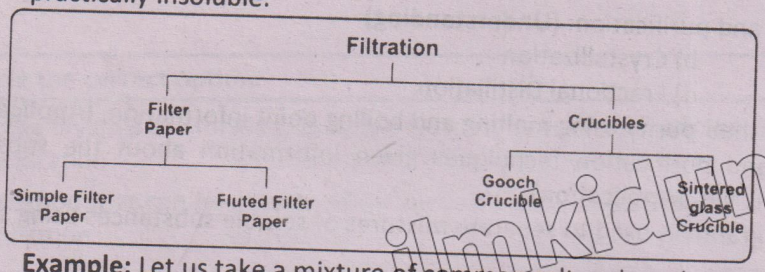




Figure: The process of filtration

- **Example:** Let us take a mixture of common salt and sand.
- Dissolve this mixture in distilled water. The common salt will distribute itself throughout water to give a homogeneous mixture. This homogenous mixture is called a **solution** of common salt in water.
- Common salt in this solution is called a solute while water is called a **solvent**.
- In a solution, the solvent is that component which is present in excess as compared to the solute.
- In this process, common salt disappears in water, the other component, sand remains undissolved and after some time settles down at the bottom of solution.
- The process of filtration is used to separate sand from the above solution.

Rack Your Mind!

1. What is filtration?

Rack Your Mind!

2. What is the residue in filtration?
   a) Liquid collected after filtration
   b) Solid left on filter paper
   c) Solvent
   d) Solute

- **Use of Filter media:** It can be performed with several types of filter media.
- Which filter medium will be used is decided on the basis of nature of the precipitate and other factors.
- The most convenient ways of filtration are either through a filter paper or through a filter crucible.

a) Filtration by using Filter Paper

Folding of filter paper is important and is done in two ways.

1. **Filtration by a Glass Funnel and Filter Paper:** It is usually a slow process. As the mixture is poured onto the filter paper, the solvent (water) passes through leaving behind the suspended particles on the filter paper. 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. The size of filter paper should be large enough so that it is one-fourth to one-half full of precipitate at the end of filtration. In this way, the filtrate runs down the side of beaker without splashing. A complete filtration assembly is shown in Figure.

Rack Your Mind!

3. What is Porosite?

Rack Your Mind!

4. The word porosity means:
   (A) Size of pores
   (B) Number of pores
   (C) Quality of pores
   (D) Nature of pores

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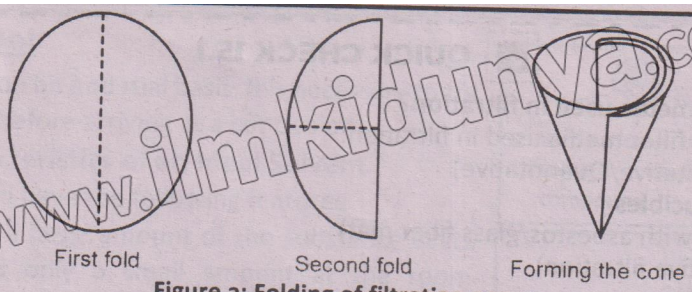


Figure a: Folding of filtration paper

The folded filter paper may then be inserted into 60° funnel, moistened with water and firmly pressed down.

2. **Fluted Filter Paper:** The rate of filtration through conical funnel can be considerably increased using a Fluted Filter Paper. For preparation of such a paper ordinary filter paper in folded in such a way that a fan like arrangement with alternate elevations and depressions at various folds is obtained Fig (b).

Rack Your Mind!

5. Why fluted filter paper is used for greater rate of filtration than ordinary cone filter paper?

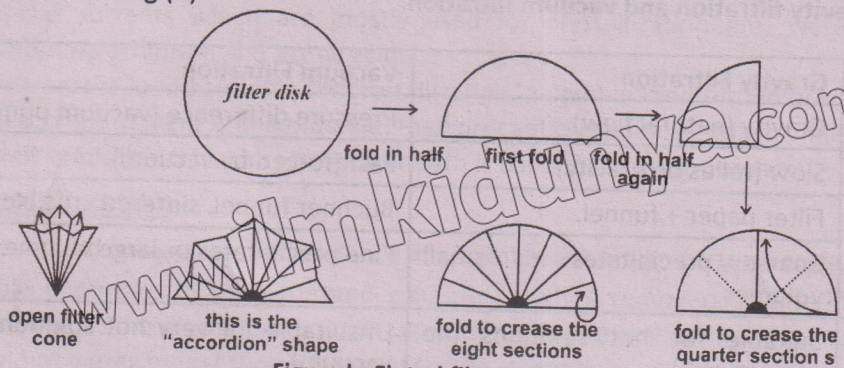


Figure b: Fluted filter paper

After filtration, the liquid which is obtained in the conical flask underneath the funnel is called the filtrate. The sand left behind on the funnel is called the residue.

Rack Your Mind!

6. Rate of filtration can be increased by using:
   (A) Desiccator (B) Glass funnel
   (C) Cold finger (D) Suction flask

b) Filtration by using Crucible

Another convenient way to filter a precipitate is by suction through a crucible. Two types of crucibles are generally used.

1. **Gooch crucible** is made of porcelain having a perforated bottom which is covered with paper pulp or a filter paper cut to its size (as shown in Figure). Quick filtration can be done by placing such crucible in a suction filtering apparatus. It is useful for the filtration of precipitates, which need to be ignited at high temperature. If its perforations are covered with asbestos mat, it may be used to filter solutions that react with paper e.g., concentrated $HC\ell$ and $KMnO_4$ solutions.

Rack Your Mind!

7. Gooch crucible is made of:
   (A) Glass (B) Paper
   (C) Teflon (D) Porcelain



Figure: Gooch crucible



Figure: Sintered glass crucible

2. **Sintered glass** crucible is a glass crucible with a porous glass disc sealed into the bottom. It is very convenient to use because no preparation is needed as with the Gooch crucible as shown in Figure.

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QUICK CHECK 15.1

a) Name different filter media used in filtration?

Ans. Here are the common filter media used in filtration:

1. **Filter Paper** (Qualitative/Quantitative)
2. **Sintered Glass Crucibles**
3. **Gooch Crucibles** (with asbestos/glass fiber mat)
4. **Charcoal** (Adsorptive filtration)
5. **Cotton or Wool Plugs**

b) What are the components of the filtration process?

Ans. The components of the filtration process are:

1. **Filter Medium** (e.g., paper, sintered glass).
2. **Slurry/Solution** (mixture to be filtered).
3. **Filtrate** (liquid that passes through).
4. **Residue/Precipitate** (solids retained).
5. **Driving Force** (gravity, vacuum, or pressure).

c) Differentiate gravity filtration and vacuum filtration.

Ans.

Feature	Gravity Filtration	Vacuum Filtration
Driving Force	Gravity (natural flow).	Pressure difference (vacuum pump/suction).
Speed	Slow (relies on gravity).	Fast (forced by vacuum).
Apparatus	Filter paper + funnel.	Büchner funnel, sintered crucible + vacuum flask.
Use Case	Coarse precipitates or small volumes.	Fine precipitates or large volumes.
Heat Compatibility	Suitable for hot solutions (no vacuum risk).	Unsuitable for very hot solutions (boiling under vacuum).
Precision	Less efficient for fine particles.	Better for fine/quantitative separations.

d) Differentiate between Gooch and sintered glass crucible.

Ans. Gooch Crucible:

- It is made up of porcelain.
- It has 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 crucibles in a suction filtering apparatus.

Advantages:

- It is useful for the filtration of precipitates, which need to be ignited at higher temperature.
- If its perforations are covered with asbestos mat then it may be used to filter solutions that react with paper e.g. concentrated $HC\ell$, $KMnO_4$ (alkaline) solutions etc.

Sintered Glass Crucible:

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

Advantages:

- There is no need to place any kind of filter paper in its bottom.
- There is no contamination of filter paper when we collect the residue. No preparation is needed as with the Gooch crucible.

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CRYSTALLIZATION

(Exercise L.Q.4)

Q. Describe the criterion to choose the suitable solvent in the process of crystallization?

Definition:

The basic principle of crystallization is the fact that 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. The process of crystallization involves the following steps:

Rack Your Mind!

8. How is crystallization used for purification?

➲ Choice of a Solvent

The solvent is chosen on hit and trial basis. It is necessary to try a number of solvents before arriving at a conclusion.

Rack Your Mind!

9. Write down the main characteristics of a solvent selected for crystallization of a compound.

➢ Properties or Characteristics of an Ideal Solvent

An ideal solvent should have the following features.

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

KEEP IN MIND

During crystallization, sometimes the crystals start to appear during the process of filtration. This result in choking the filter paper or the funnel. To avoid this a hot water funnel may be used.

Solvent Used: The solvents which are mostly used for crystallization are, water, absolute ethanol, chloroform, carbon tetrachloride and acetic acid.

If none of the solvents is found suitable for crystallization, a combination of two or more miscible solvents may be employed. If the solvent is inflammable, then precaution should be taken while heating the solution so that it does not catch fire. In such cases, water bath is used for heating purpose.

➲ Steps of Crystallization

- Prepare a saturated solution of the substance in a suitable solvent at the temperature of the experiment.
- The insoluble impurities in the saturated solution are then removed by filtering the hot saturated solution. This avoids the premature crystallization of the solute on the filter paper or in the funnel stem. If necessary, hot water funnel should be used for this purpose.
- The hot filtered solution is then cooled at a moderate rate so that medium sized crystals are formed.

KEEP IN MIND

Sometimes during the preparation of a crude substance, the colouring matter or resinous products affect the appearance of product. Such impurities are conveniently removed by boiling the substance in the solvent with the sufficient quantity of finely powdered animal charcoal and then filtering the hot solution. The coloured impurities are adsorbed by animal charcoal and the pure decolourized substance crystallizes out from the filtrate on cooling.

- When the crystallization is complete, the mixture of crystals and the mother liquor is filtered through a Gooch crucible using a vacuum pump.

Drying the Crystals:

- The crystals may be dried in the following ways:

(i) Air-dried: The crystals may be air dried.

(ii) Drying in an Oven: The crystals are dried in an oven provided the substance does not melt or decompose on heating at 100°C.

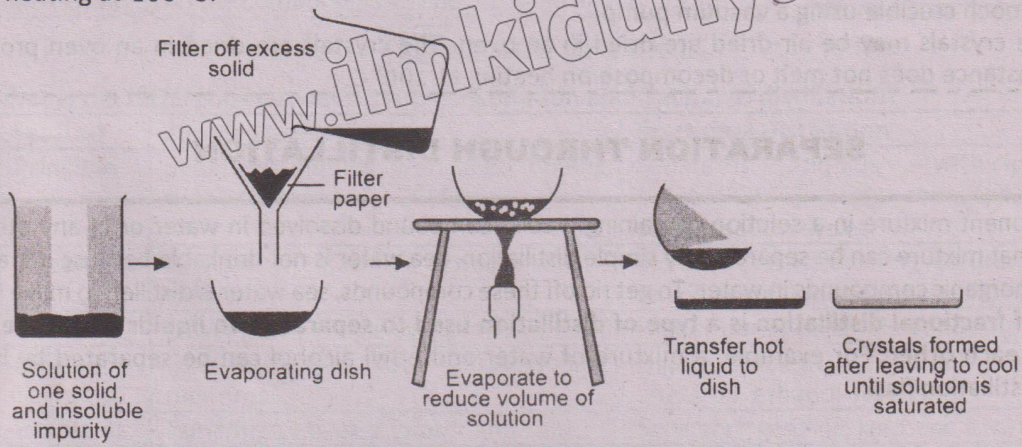


Figure: Different steps involved in crystallization

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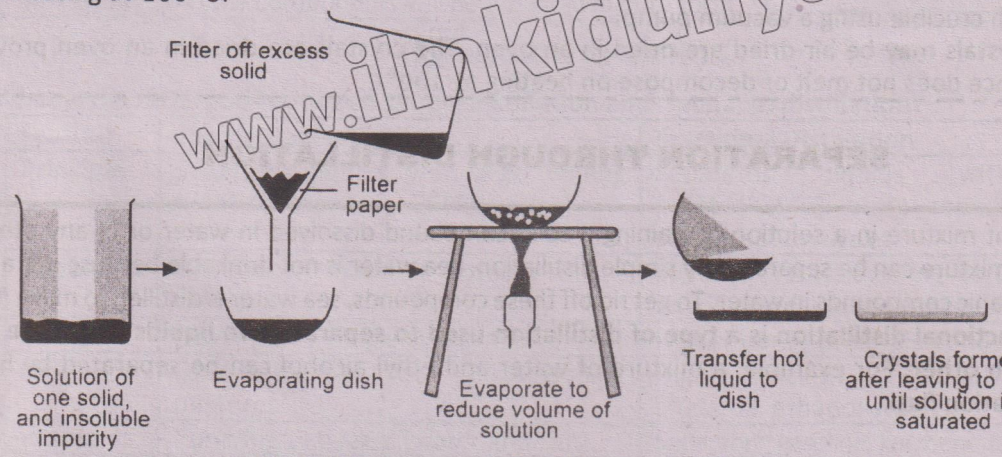


Figure: Different steps involved in crystallization

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(iii) Drying through Vacuum Desiccator:

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

Drying Agents: The drying agents used in a desiccator are $CaCl_2$, silica gel or phosphorus pentaoxide.

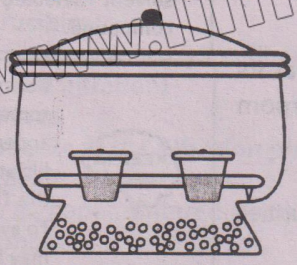


Figure: Vacuum Desiccator

Q. What is safe and reliable method of drying the crystals?

QUICK CHECK 15.2

a) What is the basic principle of crystallization?

Ans. Basic Principle of Crystallization:

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

b) How a suitable solvent is selected for the process of crystallization?

Ans. Choice of a Solvent

The solvent is chosen on hit and trial basis. It is necessary to try a number of solvents before arriving at a conclusion.

Properties or Characteristics of an Ideal Solvent

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

c) Mention the important steps of crystallization.

Ans. Important Steps of Crystallization:

i. Prepare a saturated solutions of the substance in a suitable solvent at the temperature of the experiment.

ii. The insoluble impurities in the saturated solution are then removed by filtering the hot saturated solution. This avoids the premature crystallization of the solute on the filter paper or in the funnel stem. If necessary, hot water funnel should be used for this purpose.

iii. The hot filtered solution is then cooled at a moderate rate so that medium sized crystals are formed.

iv. When the crystallization is complete, the mixture of crystals and the mother liquor is filtered through a Gooch crucible using a vacuum pump.

v. The crystals may be air-dried are dried in an oven. The crystals are dried in an oven provided the substance does not melt or decompose on heating at 100°C.

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SEPARATION THROUGH DISTILLATION

If a two-component mixture in a solution containing a solid compound dissolved in water or in any other suitable solvent, then that mixture can be separated by simple distillation. Sea water is not drinkable because it is a mixture of many soluble inorganic compounds in water. To get rid off these compounds, sea water is distilled to make it drinkable. **The process of fractional distillation is a type of distillation used to separate two liquids which are soluble or miscible with each other.** For example, a mixture of water and ethyl alcohol can be separated by heating the mixture in a distillation flask.

➲ Distillation Process

S.O. Differentiate between distillate and residue.

1. Simple Distillation

It is used to separate solvent from solution.

Example: Take 100 cm^3 sea water. Set up the apparatus as shown in the Figure. Add a few pieces of boiling chips to prevent bumping of the liquid in the flask. Make whole apparatus air tight to prevent vapors to escape.

Heat the flask gently with the help of a Bunsen burner. Turn on the tap to allow cold water to circulate slowly around the condenser. Water or alcohol will evaporate as its boiling point is reached, and its vapours will pass through the condenser. The water circulating around the condenser will condense these vapours back into the liquid form. This is called **distillate** and it will be collected in the receiving flask. The components left behind in the distillation flask are collectively called the **residue**.

2. Fractional Distillation

Separating the two miscible liquids in this way is called fractional distillation. The process of fractional distillation will be successful only if the difference in the boiling points of the two liquids being separated, is around 25°C. The distillation column is a glass equipment used in the distillation of mixture of liquids to separate it into its components depending upon their boiling points. The distillation column is filled with glass beads to increase the surface area available for condensing the vapor as shown in Figure.

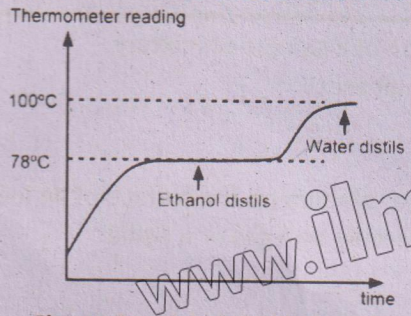


Figure: Fractional distillation of ethanol / water mixture

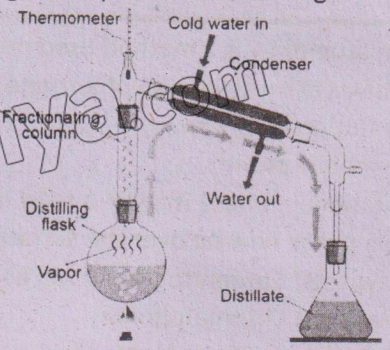


Figure: Fractional distillation setup

Separation of a Mixture of Ethyl Alcohol and Water:

Example:

- When a mixture of ethyl alcohol and water is heated, first ethyl alcohol will be distilled over because its boiling point is 78°C.
- Which is less than that of water whose boiling point is 100°C.
- As long as the alcohol is being distilled over, the temperature of the thermometer will remain at 78°C.
- As soon as the temperature starts rising above 78°C, replace the receiving flask with a new one as shown in Figure 15.7.
- Start heating again. When the temperature of the thermometer rises to 100°C, water will start boiling and is collected in the receiving flask after condensation as the second distillate.
- In this way both the components will be obtained in pure form.

Interesting Information!

Petroleum in natural form is a mixture of many compounds which are commonly used as fuels. Example of such fuels are petrol, diesel, kerosene oil and furnace oil. These components have boiling points not very different from one another. These components are separated as fractions by fractional distillation using a fractionating or distillation column.

☑ QUICK CHECK 15.

a) What is the difference between simple distillation and fractional distillation?

Ans.

Feature	Simple Distillation	Fractional Distillation
Principle	Separates liquids with **large boiling point differences** (≥25°C).	Separates liquids with **close boiling points** (<25°C) using repeated vaporization-condensation cycles.
Apparatus	Distillation flask + condenser + receiver.	Includes a **fractionating column** (packed with beads/plates) for enhanced separation.
Purity	Lower purity (single evaporation step).	Higher purity (multiple condensation steps).
Efficiency	Less efficient for similar-boiling mixtures.	Highly efficient for complex mixtures (e.g., crude oil, ethanol-water).
Examples	Purifying seawater (water and salt).	Separating gasoline, kerosene, etc., from crude oil.

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b) Give some daily-life applications of fractional filtration/distillation?

Ans. Applications:

1. **Petroleum Refining**
   - Separates crude oil into gasoline, diesel, kerosene, and lubricants.
   - Each fraction has different boiling points (e.g., gasoline evaporates first).
2. **Alcohol Separation**
   - Ethanol (78°C) and water (100°C) are close-boiling; fractional distillation achieves high purity.
3. **Essential Oils & Perfumes**
   - Extracts fragrant compounds from plants.
4. **Air Separation**
   - Produces liquid oxygen, nitrogen, and argon from air. Gases have slightly different boiling points (−183°C to −196°C).
5. **Water Purification**
   - Removes volatile contaminants in labs/industries.

CHROMATOGRAPHY

- Chromatography is a method used primarily for the separation of a sample of mixture.
- It involves the distribution of a solute (mixture) between two phases:
  (i) a stationary phase
  (ii) a mobile phase
- The stationary phase may be a solid or a liquid supported as a thin film on the surface of an inert solid. The mobile phase flowing over the surface of the stationary phase may be a gas or a liquid.
- Two types of Chromatography will be discussed here:
  (i) Partition Chromatography
  (ii) Adsorption Chromatography

Differences between Adsorption and Partition Chromatography

Partition Chromatography	Adsorption Chromatography
• The chromatography in which the stationary phase is a liquid is called partition chromatography.	• The chromatography in which the stationary phase is a solid is called adsorption chromatography."
• In this type, the substances being separated are distributed throughout both the stationary and mobile phases.	• In this type, a substance leaves the mobile phase to become adsorbed on the surface of solid stationary phase.
Examples • Paper chromatography • Gas liquid chromatography	**Examples** • Column chromatography • Thin layer chromatography • Gas solid chromatography

(Exercise L.Q.5)

Q. What is the basic principle of paper chromatography? Give its three practical life applications.

➲ Paper Chromatography

- Paper chromatography is a type of partition chromatography.

Stationary and mobile phases in paper chromatography:

- The entrapped water in cellulose fibers of paper is stationary phase and mobile phase which passes over the paper are immiscible, the mobile phase is usually an organic liquid.

Types of Paper Chromatography:

- There are three common ways of carrying out paper chromatography namely:
  (i) ascending (ii) descending (iii) radial/circular.
- Only the ascending type will be discussed here. In this technique, the solvent travels upwards by capillary action.

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Procedure of Ascending Paper Chromatography:

A solvent mixture, specially composed in accordance with the sample to be separated, is poured into the chromatographic tank Figure. Cover the tank to homogenize its inner atmosphere. 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. Spot a point, on the pencil line, with the sample mixture solution. To facilitate identification of the components of the mixture, spots of the known compounds may also be placed along side.

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. Cover the tank. As the solvent front passes the spots, the solutes begin to move upward. Different components of solute will move at different rates. This separates the mixture. When the solvent front has risen to about length of the paper, remove the strip, mark the solvent front with a pencil and allow the strip to dry.

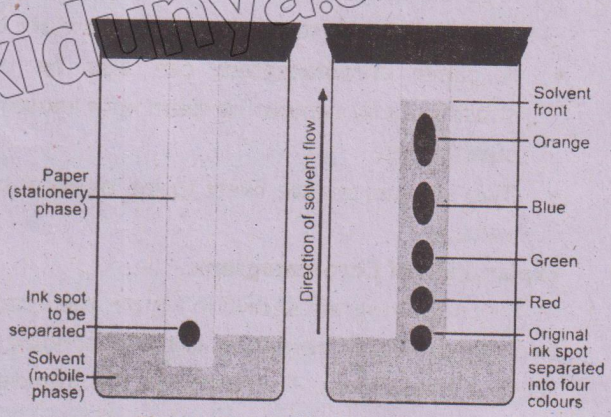


Figure: Paper Chromatography

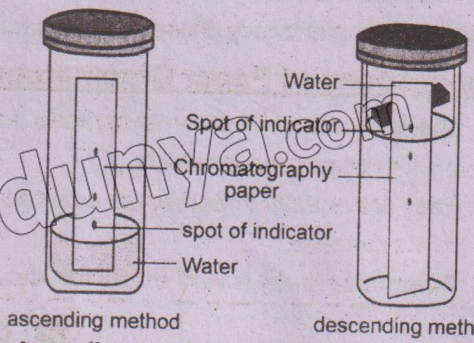


Figure: Ascending and Descending Paper Chromatography

Locating Agents for Colorless Substances

Once the paper is dried, the pattern on the paper is called a **chromatogram**. The different components of the mixture, if coloured, can visually be identified. If colourless, the chromatogram has to be developed by chemical methods or physical techniques used to identify the spots.

Use of a UV Lamp or Light: A very convenient way of visualizing the colourless chromatogram is to see it under the light of a UV lamp. The spots look coloured under UV light. Alternatively, a locating agent like ninhydrin can be used to locale the spots.

Locating Agent: A locating agent is generally a chemical that reacts with the colourless substances like amino acids to give coloured products that are visible for inspection.

Use of Retardation Factor

Each component has a specific retardation factor called R_f value. The R_f value is given by:

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

With reference to Figure the chromatogram shows that the sample A contains both components B and C. The R_f values for B and C are given by:

$$R_f(B) = \frac{x}{y}$$

$$R_f(C) = \frac{x}{y}$$

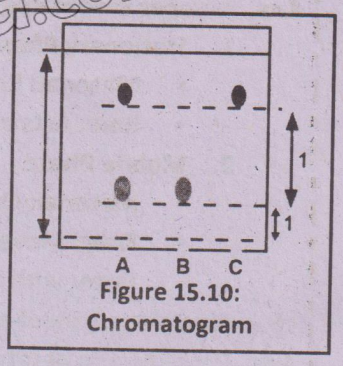


Figure 15.10: Chromatogram

Use/Significance of Paper Chromatography:

- Paper chromatography is a convenient way of checking if a substance formed in a chemical reaction is pure or impure using a suitable solvent in which the substance dissolves.

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- A pure substance produces one spot on the chromatogram while an impure substance produces more than one spot.
- A paper chromatogram can also be used to identify substances by comparing them with known samples of pure substances.
- Two substances are likely to be identical have the same R_f values.

Explanation of Chromatogram:

In this chromatogram shown in Figure, the impure substance has two components present in it corresponding to two coloured spots it has shown. The spots in the impure substance have travelled the same distance as the reference's pure compounds. The R_f value is always the same for a particular substance if we use the same stationary phase and the same mobile phase.

Interesting Information!

Whatmann filter paper which are used in paper chromatography are made from specially selected cotton cellulose. Whatmann's filter paper No. 1 is used for routine applications and it has medium retention and medium flow rate. Its pore size is 11 μm.

Rack Your Mind!

10. Which of the following is used to identify unknown substances in paper chromatography?

a) Solvent front b) R_f value
c) Residue d) Filtrate

Applications of Paper Chromatography

i. The techniques of chromatography are very useful in organic synthesis for separation, isolation and purification of the products.
ii. They are equally important in qualitative and quantitative analyses and for determination of the purity of a substance.
iii. Chromatography is very useful for identifying complicated chemicals such as dyes, drugs and pesticides.

QUICK CHECK 15.4

a) On which basis the components of a mixture are separated from one another in paper chromatography?

Ans. In paper chromatography, components of a mixture are separated based on their differential affinities for the two phases involved:

1. **Stationary Phase**:
   - **Water molecules** bound to cellulose fibers in the paper (polar/hydrophilic).
   - **Polar compounds** interact strongly (slow movement).
2. **Mobile Phase**:
   - **Solvent** (e.g., ethanol, acetone) moving via capillary action.
   - **Non-polar compounds** dissolve better in the solvent (fast movement).

b) What are the stationary and mobile phases in paper chromatography?

Ans. In **paper chromatography**, the two phases are:

1. **Stationary Phase**
   - **Material:** Cellulose fibers in the filter paper + **adsorbed water** (from humidity or solvent).
   - **Role:** Acts as a **polar/hydrophilic** layer. Polar compounds bind to it (slow movement).
2. **Mobile Phase**
   - **Material:** Organic solvent or solvent mixture (e.g., ethanol, acetone, water-methanol).
   - **Role:** Moves via capillary action, carrying dissolved compounds. Non-polar compounds travel faster with it.

c) How the purity of a substance is determined by using paper chromatography?

Ans. Procedure to determine the **purity of a substance** using paper chromatography:

1. **Spot the Sample:**
   - Apply a small drop of the substance on the chromatography paper.
2. **Develop the Chromatogram:**
   - Immerse the paper in a solvent (mobile phase). Allow the solvent to rise via capillary action.

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3. **Analyze the Results:**
   - **Pure Substance:** Forms **a single spot** (all molecules have identical R_f values).
   - **Impure Substance:** Shows **multiple spots** (each spot = a different component).
4. **Compare R_f Values:**

Calculate

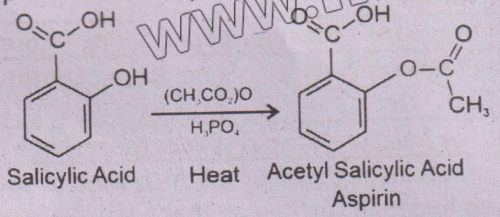
- R_f = Distance traveled by spot (Distance travelled by solvent).
- Pure compounds have a **consistent R_f**, impurities show additional spots with different R_f.
  - **Single spot** = High purity.
  - **Multiple spots** = Contaminants present.
  - **Confirmation:** Repeat with different solvents for reliability.

HOW TO CHECK THE PURITY OF THE PRODUCT?

To Purify Compounds:

Separate methods are applied to purify compounds if they exist in liquid or solid states under normal conditions. Liquids are generally purified by distilling them at normal pressure or under vacuum. Purity of liquids can be checked by comparing their boiling points with those mentioned in the literature.

Preparation of Aspirin: Aspirin is prepared by reacting salicylic acid with an excess of acetic anhydride in the presence of a few drops of concentrated H_2SO_4. Aspirin is a very important analgesic and antipyretic. After the reaction is over, water is added in the reaction mixture to precipitate out aspirin. The mixtu e is then filtered to get aspirin as a crude product. It is then crystallized in water as polar solvent.



Salicylic Acid — Heat — Acetyl Salicylic Acid (Aspirin)

Rack Your Mind!

11. What type of reaction is involved in the synthesis of Aspirin?

i. Aspirin obtained in the above process may be mixed with the unreacted starting material, salicylic acid.

To Check Whether the Product is Pure or Impure:

One way of assessing the purity is its melting point is determined. If the product melts sharply, e.g. Salicylic Acid melts at 136°C then the product is considered as pure. If the product shows a melting point which is not sharp i.e. it melts within a range of temperature, it means it is an impure product.

ii. Other way of checking the purity of the product is to get the mixed melting point. In such a case the product is mixed with a pure sample of aspirin and the melting point is then determined. If it still shows a sharp melting point then it is a pure product.

iii. Another way of checking the purity of aspirin is to run paper chromatography of this sample along with the pure samples of both aspirin and salicylic acid and matching their R_f value.

☑ QUICK CHECK 15.5

a) How does melting point of a substance indicate its purity?

Ans. The melting point indicates purity as follows:

1. **Pure Substance**:
   Melts at a sharp, specific temperature (e.g., pure aspirin melts at 135°C).
2. **Impure Substance**:
   Melts over a broader, lower range (e.g., 120–130°C) due to disrupted crystal structure.

b) Which technique (filtration, crystallization and distillation) is suitable for the separation of the following mixtures?

i) **Petroleum and water**
ii) **NaCl and $NaNO_3$**
iii) **activated charcoal and sodium chloride**
iv) **Methanol, ethanol and propanol**

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Ans.

i) **Petroleum and water**
- **Technique: Separating funnel** (immiscible liquids) or decantation.
- Petroleum (non-polar) and water (polar) form distinct layers.

ii) **NaCℓ and $NaNO_3$**
- **Technique:** Fractional crystallization.
- Differing solubilities in water allow selective crystallization (e.g., $NaNO_3$ crystallizes first on cooling).

iii) **Activated charcoal and sodium chloride**
- **Technique: Filtration** (charcoal is insoluble).
- Charcoal particles are trapped on filter paper; NaCℓ solution passes through.

iv) **Methanol, ethanol, and propanol**
- **Technique:** Fractional distillation.
- Their boiling points (methanol: 65°C, ethanol: 78°C, propanol: 97°C) are close but separable via a fractionating column.

Solution File — Rack Your Brain!

Sr. #	Option	Explanation
1.	S.Q	Filtration is a method used to separate an insoluble solid from a liquid by passing the mixture through filter paper. • The solid trapped on filter paper is called **residue**. • The liquid passing through is called **filtrate**.
2.	B	Residue is the insoluble solid particles trapped on the filter paper during filtration.
3.	S.Q	Porosites means pore sizes. It is related to porosity which is a measure of empty spaces within a solid material.
4.	A	Porosity is commonly used. Porosity is the measure of void spaces (pores) in a material.
5.	S.Q	The rate of filtration through conical funnel can be considerably increased by using fluted filter paper. For this an ordinary filter paper is folded in such a way that a fan like arrangement with alternate elevations and depressions at various folds is obtained. In this way, we increase the surface area of filter paper. As a result, the rate of filtration increases.
6.	D	Rate of filtration can be increased by using Suction flask an instrument used for vacuum filtration.
7.	D	Gooch crucible is made of porcelain having a perforated bottom which is covered with paper pulp or a filter paper cut to its size.
8.	S.Q	Crystallization separates pure solid crystals from impurities. The impure solid is dissolved in hot solvent; upon cooling, pure crystals form and can be collected by filtration.
9.	S.Q	See Topic Choice of a Solvent (Properties or Characteristics of an Ideal Solvent).
10.	B	R_f value helps compare the movement of an unknown substance with known substances for identification.
11.	S.Q	Esterification (See Reaction in synthesis of Aspirin).

Exercise

MULTIPLE CHOICE QUESTIONS (MCQs)

Q.1 Four choices are given for each question. Select the correct choice.

I. After the crystals are formed, they are typically separated from the mother liquor by:

a) Evaporation
b) Decantation or filtration
c) Sublimation
d) Distillation

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II. **The comparative rates at which the solutes move in paper chromatography depend on:**
a) The size of filter paper
b) R_f values of solutes
c) Temperature
d) Size of the chromatographic jar

III. **The method that can be used to separate two solid compounds with different solubilities in a solvent is:**
a) Distillation
b) Isolation
c) Crystallization
d) Filtration

IV. **It is suspected that a hand-written legal document has been changed by over writing some crucial figures. Which technique you will use to check the inks used at suspected places?**
a) Distillation
b) Chromatography
c) Solvent Extraction
d) Crystallization

V. **In chromatography, the components of the mixture are separated based on:**
a) Their molecular masses
b) The interaction of the components with stationary as well as mobile phone
c) The type of solvent used
d) The filter paper used

VI. **The porous material used to separate the solid from the liquid during filtration is called the:**
a) Filtrate
b) Residue
c) Filter medium
d) Solvent

VII. **The key difference between simple distillation and fractional distillation is the presence of a:**
a) Condenser with a larger surface area.
b) More powerful heat source.
c) Fractionating column.
d) Vacuum pump.

VIII. **The essential requirement for separating two liquids by simple distillation is that their boiling points should differ by at least:**
a) 5°c
b) 10°C
c) 25°C
d) 50°C

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Answer Key with Explanations

Sr.No.	Option	Answer	Explanation
I.	b	Decantation or filtration	• After crystallization, crystals are separated from the mother liquor (solution) by filtration (using filter paper) or decantation (pouring off the liquid).
II.	b	R_f values of solutes	• In paper chromatography, solute movement depends on R_f (retardation factor), which is specific to each compound's affinity for the stationary/mobile phases.
III.	c	Crystallization	• Crystallization exploits differences in solubility to separate solids. The less soluble compound crystallizes first, while the other remains in solution.
IV.	b	Chromatography	• Chromatography (e.g., TLC or HPLC) can distinguish between inks by separating their components based on differing chemical properties.
V.	b	The interaction of the components with stationary as well as mobile phone	• Separation occurs due to differential affinity of components for the stationary phase (e.g., filter paper) vs. the mobile phase (solvent).
VI.	c	Filter medium	• The filter medium (e.g., filter paper, sintered glass) traps solids while allowing liquids (filtrate) to pass through.
VII.	c	Fractionating column	• Fractional distillation uses a fractionating column to provide multiple vaporization condensation cycles, improving separation of liquids with close boiling points.
VIII.	c	25°C	• Simple distillation requires a minimum 25°C difference in boiling points for effective separation. Smaller differences need fractional distillation.

SHORT ANSWER QUESTIONS

Q.2 Attempt the following short-answer questions:

a. Why is there a need to crystallize a crude product?

Ans. The preparation of chemical compounds usually affords a crude product. The crude product may contain soluble and insoluble impurities. Insoluble impurities are removed by filtration while soluble impurities are removed by crystallization process. Crystallization also gives a definite geometrical shape to the crystals of the crude product. So in order to get pure product in the form of crystals there is a need to crystallize the crude product.

b. What is the function of fluted filter paper?

Ans. The **function of fluted filter paper** is to **increase the filtration efficiency** by providing a larger surface area and faster flow rate compared to flat filter paper.

Functions (Features):

1. **Faster Filtration**
   - The fluted (folded) design creates channels for the liquid to flow through, reducing clogging and speeding up the process.
2. **Increased Surface Area**
   - More folds = more contact points for solids to collect, preventing premature blockage.
3. **Better Airflow**
   - Gaps between folds allow air to escape, preventing pressure buildup that can slow filtration.

c. What is the difference between a Gooch crucible and sintered glass crucible?

Ans.

Feature	Gooch Crucible	Sintered Glass Crucible
Material	Porcelain or silica, lined with asbestos/glass fiber mat.	Made entirely of fused glass with porous frit.
Porosity	Adjustable (depends on mat used).	Fixed porosity.
Use Case	Filtering fine/gelatinous precipitates (e.g., $BaSO_4$).	Filtering fine to coarse precipitates (e.g., $AgC\ell$).
Heat Resistance	High (can be ignited/dried at high temps).	Moderate (may crack with rapid temp changes).
Chemical Resistance	Resists acids/bases (depends on mat).	Resists most acids (except HF) but not strong alkalis.
Cleaning	Mat must be replaced; crucible reused.	Reusable; cleaned with acids or solvents.
Typical Setup	Used with vacuum filtration.	Used with vacuum or gravity filtration.

d. What type of mixtures are filtered through a Gooch crucible?

Ans. Gooch crucibles are used to filter **precipitate-solid and liquid mixtures**, especially **gravimetric analysis samples** where the solid is dried and weighed.

e. What is function of fractionating column during fractional distillation?

Ans. The fractionating column in fractional distillation provides multiple vaporization-condensation cycles, enhancing separation of liquids with close boiling points by increasing surface area for selective condensation of higher-boiling components.

- **Improves purity** by allowing repeated condensation and re-vaporization.
- **Enables separation** of mixtures (e.g., crude oil into gasoline, kerosene, etc.).

f. What is the stationary phase in the paper chromatography?

Ans. In paper chromatography, the stationary phase is the water molecules adsorbed (bound) onto the cellulose fibers of the filter paper.

1. **Composition:**
   - Cellulose (paper) + bound water (from humidity or solvent).
   - Acts as a polar hydrophilic layer.
2. **Function:**
   - Interacts with analytes via hydrogen bonding and dipole forces.
   - Polar compounds bind more strongly, moving slower; non-polar compounds move faster.

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g. What will happen during paper chromatography if the components of the mixture have a comparable attraction for the stationary phase?

Ans. If the components of a mixture have **comparable attraction for the stationary phase** in paper chromatography:

1. **Poor Separation:**
   - Components will travel at **similar rates**, staying close together on the paper.
2. **Overlapping Spots:**
   - Bands/spots may **merge**, making it difficult to distinguish individual compounds.
3. **Low Resolution:**
   - R_f values (retention factors) will be **nearly identical**.

Solution:
Use a different **mobile phase** or modify the stationary phase (e.g., polar/non-polar solvents) to alter affinities.

h. What is the meant by the term "developing the chromatogram" in paper chromatography?

Ans. **"Developing the chromatogram"** in paper chromatography refers to the process of **allowing the mobile phase (solvent) to migrate up the paper**, separating the mixture's components based on their differential affinities for the stationary (paper) and mobile phases.

Important Points:

- **Solvent Movement:** The mobile phase travels along the paper via capillary action.
- **Separation**: Components partition between the stationary (cellulose/water) and mobile phases, forming distinct spots/bands.
- **Completion**: The process stops when the solvent front nears the top of the paper.

i. What is the basic principle of paper chromatography?

Ans. The basic principle of paper chromatography is differential partitioning of components between two phases:

- **Stationary phase**: Water-bound cellulose fibers in the paper (polar/hydrophilic).
- **Mobile phase**: Solvent (e.g., ethanol, water) moving via capillary action.

j. Why water is not generally used as a solvent in paper chromatography?

Ans. Water is rarely used alone in paper chromatography because:

- **Slow Evaporation:** Prolongs drying time.
- **Poor Solubility:** Many organic compounds dissolve poorly in water.
- **Weak Separation:** Limited ability to differentiate components due to high polarity.

k. Differentiate between adsorption and partition chromatography.

Ans. **See** (Differences between Adsorption and Partition Chromatography) **{Already Answered above}**

l. How can you check the purity of a compound with the help of paper chromatography?

Ans. **To check the purity of a compound using paper chromatography:**

1. **Run the Sample:**
   - Spot the compound on chromatography paper and develop it with a suitable solvent.
2. **Analyze the Result:**
   - **Pure Compound:** Forms **a single spot** on the chromatogram.
   - **Impure Compound:** Shows **multiple spots** (each spot = a different component).
3. **Compare R_f Values:**
   - A single R_f (Retention Factor) value confirms purity; multiple values indicate impurities.

m. You have prepared a solid sample of glucosazone in the laboratory. How will you proceed to check the purity of the sample?

Ans. **Purity of Glucosazone:**

1. **Melting Point Test:**
   Compare observed melting point with literature value (sharp range = pure).
2. **TLC (Thin-Layer Chromatography):**
   Spot on silica plate, develop solvent → **single spot** confirms purity.
3. **HPLC or NMR** (if available):
   Sharp peaks in HPLC/NMR spectra indicate purity.

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DESCRIPTIVE QUESTIONS

Q.3 Differentiate simple distillation and fractional distillation in construction and applications.

Ans. See Answer in Quick Check 15.3 (a)

Q.4 Describe the criterion to choose the suitable solvent in the process of crystallization?

Ans. **See Page No. (494)**

Q.5 What is the basic principle of paper chromatography? Give its three practical life applications.

Ans. **See Page No. (498)**

ADDITIONAL SLOs BASED MCQs

1. **Branch of chemistry that deals with the complete qualitative and quantitative analysis of a substance is:**
   A. Stoichiometry B. Physical chemistry C. Analytical chemistry D. Quantum chemistry
2. **Identification of the components of a sample is:**
   A. Quantitative analysis B. Qualitative analysis C. Stoichiometry D. Physical chemistry
3. **Which of the following technique is used for the separation of insoluble particles from liquids?**
   A. Filtration B. Crystallization C. Solvent extraction D. Chromatography
4. **The technique used to separate components of mixture in solid phase.**
   A. Crystallization B. Filtration C. Sublimation D. Solvent extraction
5. **The solid which is left over the filter paper as a result of filtration:**
   A. Filtrate B. Residue C. Precipitate D. Mother Liquor
6. **Selection of filter paper depends on size of particles to be:**
   A. Filtered B. Dried C. Decolorized D. Decanted
7. **Fluted filter paper is used to:**
   A. Filter hot solution B. Avoid premature crystallization
   C. Increase the rate of filtration D. Decrease the area
8. **NaCl and sand can be separated by one of the following techniques:**
   A. Formation of solution and filtration
   B. Formation of solution and evaporation without filtration
   C. Sublimation D. Chromatography
9. **Size of filter paper is selected according to the amount of:**
   A. Solution B. Insoluble solute C. Soluble solute D. Solvent
10. **Without suction pump, filtration is:**
    A. Fast process B. Slow process C. Rapid process D. All are possible
11. **Rate of filtration can be increased by applying gentle suction:**
    A. Gooch crucible B. Filter paper C. Sintered crucible D. All of the above
12. **The solution remaining after the formation of crystals is called:**
    A. Mother liquor B. Dilute solution C. Residue D. both 'A' and 'B'
13. **Separation of a solid from its hot saturated solution by cooling is called:**
    A. Vapourization B. Solvent extraction C. Filtration D. Crystallization
14. **Safe and the most reliable method of drying crystals is through:**
    A. Filter paper B. Vacuum desiccators C. Oven D. None of these
15. **In order to have good crystals of a substance the temperature of the system at the time of preparation of solution should be:**
    A. Around 0°C B. Around room temperature
    C. Sufficiently more than room temperature D. Just above the room temperature

Answer Key

1. C	2. B	3. A	4. C	5. B	6. A	7. C	8. A	9. B	10. B	11. D	12. A
13. D	14. B	15. C									

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ADDITIONAL SHORT ANSWER QUESTIONS

Q.1 What is Chromatography?

Ans. The technique separation in which components of a mixture of substances, (coloured pigments and coloured metallic ions) are separated on the basis of their relative affinities for stationary and mobile phases is called chromatography.

Q.2 What do you understand by the term, "chromatogram"?

Ans. The finished dried paper with the coloured bands of various components of a mixture obtained in a chromatographic experiment, is called chromatogram.

Q.3 What is the locating agent?

Ans. A suitable chemical reagent which is sprayed on the chromatogram to locate the position of various colorless substances is called locating reagent, e.g., H_2S rubeanic acid etc.

Q.4 Define analytical chemistry.

Ans. Analytical Chemistry:
"The branch of chemistry which deals with the chemical characterization (qualitative and quantitative analysis) of a compound is called analytical chemistry."

Q.5 How chemical characterization of a compound is done?
OR Write down major steps involved in complete quantitative analysis.

Ans. Four Major Steps:
Following major steps are necessary for complete quantitative analysis of a compound
(i) Obtaining a sample for analysis **(ii)** Separation of the desired constituent
(iii) Measurement and calculation of results **(iv)** Drawing conclusion from the analysis

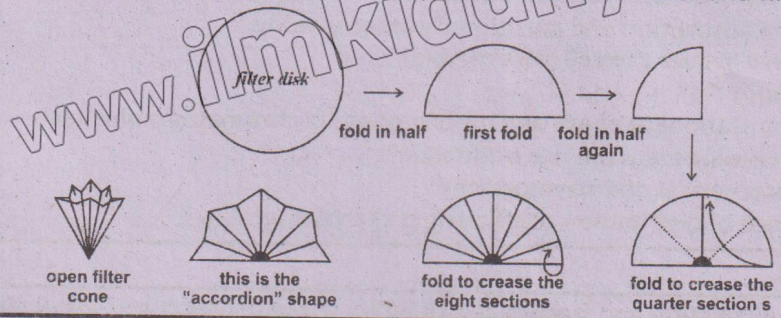
Q.6 Why fluted filter paper is used for greater rate of filtration than ordinary cone filter paper?

Ans. Fluted Filter Paper:
The rate of filtration through conical funnel can be considerably increased by using fluted filter paper. For preparation of such a 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. In this way, we increase the surface area of filter paper. As a result, the rate of filtration by fluted filter paper increases as compared to ordinary filter paper.

Q.7 Discuss folding of filter paper briefly.

Ans. Folding of Filter Paper:
The folding of filter paper is important and following points should be kept in mind:

- The paper should be folded twice. The first fold should be along the diameter of the paper. The second fold should be such that the 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 and an apex angle very slightly greater than 60°.
- The paper may then be inserted into 60° funnel, moistened with water and firmly pressed down.



Q.8 How saturated solution for crystallization can be prepared?

Ans. Preparation of the Saturated Solution:
After selecting a suitable solvent, the substance is then dissolved in a minimum amount of solvent and is heated directly or on a water bath with constant stirring.
Add more solvent to the boiling solution if necessary until all the solute has dissolved.

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Q.9 Define filtration and crystallization.

Ans. Filtration: "The process in which insoluble particles (suspended particles or precipitates) are separated from liquids is called filtration."

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

Q.10 What is safe and reliable method for drying the crystals? Briefly explain.

Ans. Vacuum Desiccator:

A **safe and reliable method** of drying crystals is through a vacuum desiccator. In this process, crystals are spread over a watch glass and kept in vacuum desiccator for several hours. **Drying agents** used in a desiccator are $CaCl_2$, silica gel or phosphorous pentaoxide (P_2O_5).

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SELF-ASSESSMENT Chapter # 15

Total Mark: 30

Q.1 Encircle the correct option. **(1 × 6 = 6)**

(i) After the crystals are formed, they are typically separated from the mother liquor by:
a) Evaporation b) Decantation or filtration c) Sublimation d) Distillation

(ii) The method that can be used to separate two solid compounds with different solubilities in a solvent is:
a) Distillation b) Isolation c) Crystallization d) Filtration

(iii) It is suspected that a hand-written legal document has been changed by over writing some crucial figures. Which technique you will use to check the inks used at suspected places?
a) Distillation b) Chromatography c) Solvent Extraction d) Crystallization

(iv) The porous material used to separate the solid from the liquid during filtration is called the:
a) Filtrate b) Residue c) Filter medium d) Solvent

(v) The key difference between simple distillation and fractional distillation is the presence of a:
a) Condenser with a larger surface area. b) More powerful heat source.
c) Fractionating column. d) Vacuum pump.

(vi) In chromatography, a locating agent is used to:
a) Dissolve the sample for separation.
b) Carry the separated components along the stationary phase.
c) Make colorless separated components visible for identification.
d) Prevent the sample from interacting with the stationary phase.

Q.2 Write short answers of the following questions. **(2 × 8 = 16)**

(i) Differentiate gravity filtration and vacuum filtration.
(ii) Differentiate between adsorption and partition chromatography.
(iii) Why HF cannot be filtered through sintered glass crucible?
(iv) What is chromatography?
(v) Differentiate between stationary phase and mobile phase in chromatographic technique?
(vi) How does a Gooch crucible increases the rate of filtration?
(vii) Write down the uses/applications of chromatography.
(viii) What is the significance of distribution coefficient in chromatography?

Q.3 Extensive Questions. **(2 × 4 = 8)**

(a) Differentiate simple distillation and fractional distillation in construction and applications.
(b) Write down the procedure of ascending paper chromatography with diagram.