15 BASIC SEPARATION TECHNIQUES

Student Learning Outcomes [C-11-E-01 to C-11-E-08]

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 is the science of chemical characterization. A complete chemical characterization of a compound must include both qualitative and quantitative analyses. In **qualitative analysis**, the chemist is concerned with the detection or identification of the elements present in a compound, whereas in **quantitative analysis**, the relative amounts of elements in compounds are determined.

A complete quantitative determination generally consists of four major steps:

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

In this chapter, some basic separation techniques are discussed here to practice in the lab.

15.1 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. **Simple distillation**
4. **Fractional distillation**
5. **Paper Chromatography**

15.1.1 Filtration

This process is used to separate one of the components of a two-component heterogeneous mixture from a solution. The first step is to select a suitable solvent in which one of the components dissolves completely while the other remains practically insoluble.

For 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.

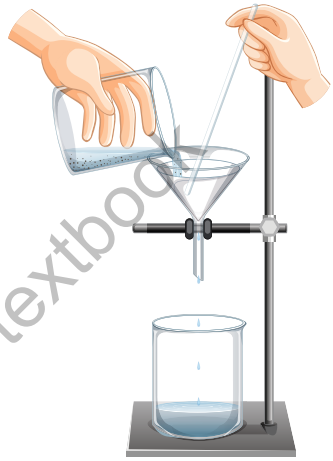


Figure 15.1 The process of filtration

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. While 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. It can be performed with several types of filter media. Nature of the precipitate and other factors dictate which filter medium must be used. The most convenient ways of filtration are either through a filter paper or through a filter crucible.

a) 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 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 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 15.1**.

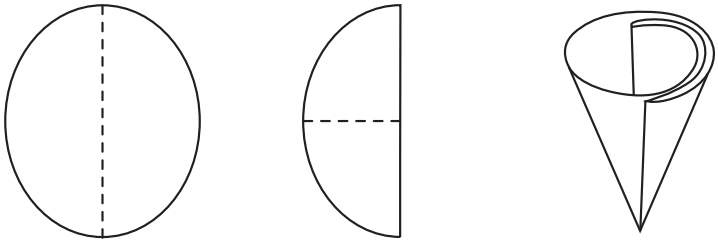


Fig 15.2 a Folding of filter paper

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

2. 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 is folded in such a way that a fan like arrangement with alternate elevations and depressions at various folds is obtained **Fig (15.2 b)**.

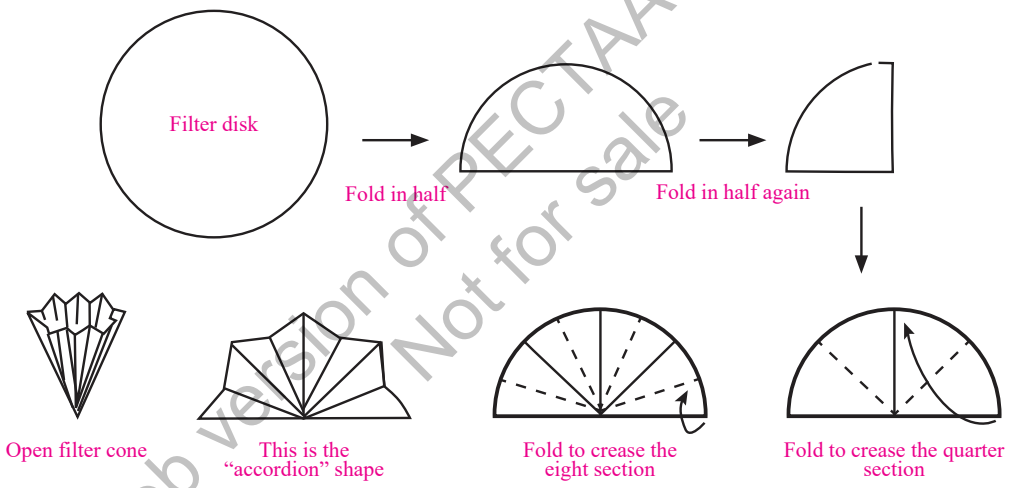


Fig 15.2 b Fluted filter paper

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**.

b) 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 15.3**. 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 may react with paper e.g. concentrated HCl and $KMnO_4$ solutions.

Figure 15.3 Gooch crucible

Fig 15.4 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 15.4**.

Quick Check 15.1

a) Name different filter media used in filtration.

b) What are the components of the filtration process?

c) Differentiate gravity filtration and vacuum filtration.

d) Differentiate Gooch and Sintered glass crucible.

15.2 CRYSTALLIZATION

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.

15.2.1 Choice of a Solvent

An ideal solvent should have the following features.

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, 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.

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.

15.2.2. Steps of crystallization

i. Prepare a saturated solution 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.

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.

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

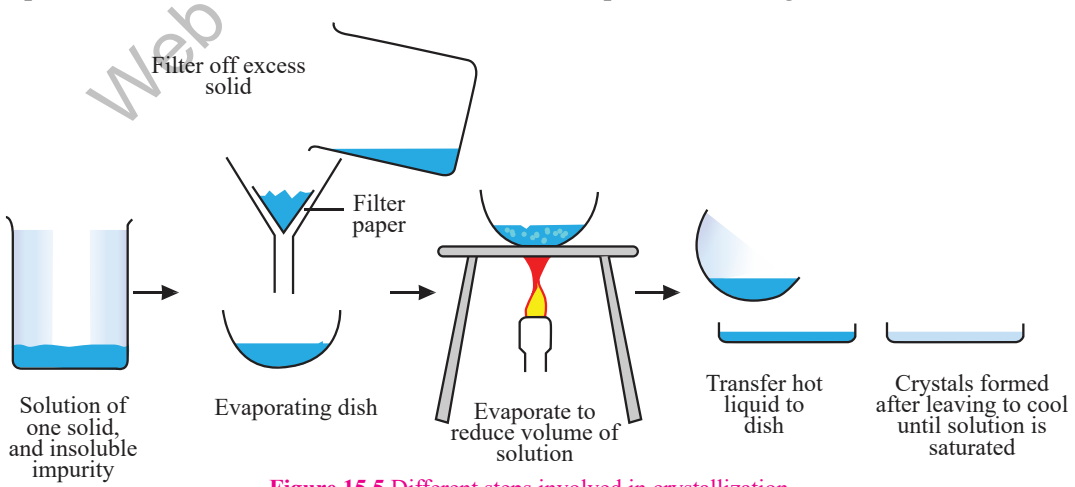


Figure 15.5 Different steps involved in crystallization

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. The drying agents used in a desiccator are $CaCl_2$, silica gel or phosphorus pentaoxide **Figure 15.5**.

Quick Check 15.2

a) What is the basic principle of crystallization?

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

c) Mention the important steps of crystallization.

15.3 SEPARATION THROUGH DISTILLATION

If a two-component mixture is 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 of these compounds, sea water is distilled to make it drinkable.

The process of **fractional distillation** is another 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.

15.3.1 Distillation Process

1. Simple distillation

It is used to separate solvent from solution. For example, take 100 cm^3 sea water. Set up the apparatus as shown in the **Figure 15.6**. 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 vapour as shown in **Figure 15.7**.

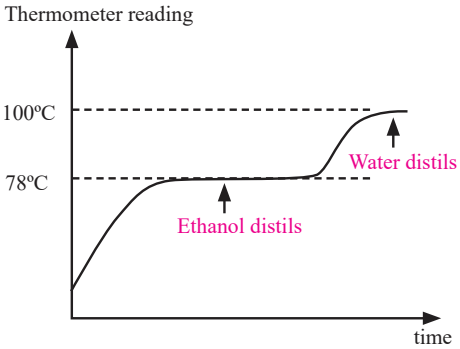


Figure. 15.6 Fractional distillation of ethanol / water mixture

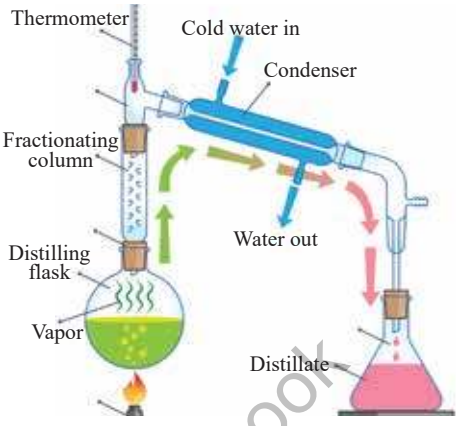


Figure 15.7 Fractional distillation setup

For 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 less than that of water whose boiling point is 100 ºC. As long as the alcohol is being distilled over, the temperature of the thermometer will remain at 78 ºC. As soon as the temperature starts rising above 78 ºC, 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 ºC, 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 15.3

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

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

15.4 CHROMATOGRAPHY

Chromatography is a method used primarily for the separation of a sample of mixture. It involves the distribution of a solute (mixture) between a stationary phase and 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.

Chromatography in which the stationary phase is a solid, is classified as **adsorption chromatography**. In this type, a substance leaves the mobile phase to become adsorbed on the surface of the solid phase. Examples of adsorption chromatography are thin layer

chromatography (TLC) and column chromatography.

Chromatography in which the stationary phase is a liquid, is called **partition chromatography**. In this type, the substances being separated are distributed throughout both the stationary and mobile phases. Examples of partition chromatography are paper chromatography and gas liquid chromatography.

15.4.1 Paper Chromatography

It is a type of partition chromatography. The entrapped water in cellulose fibers of paper and mobile phase which passes over the paper are immiscible, The mobile phase is usually an organic liquid.

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.

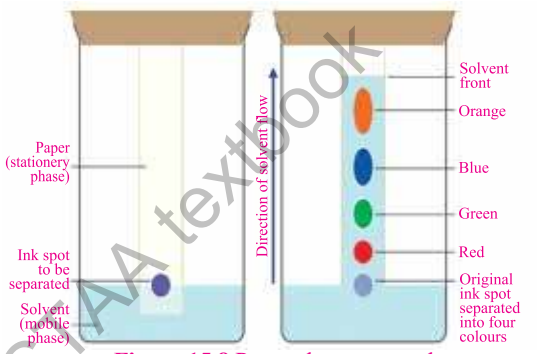


Figure 15.8 Paper chromatagraphy

A solvent mixture, specially composed in accordance with the sample to be separated, is poured into the chromatographic tank **Figure 15.8**, 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 alongside.

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.

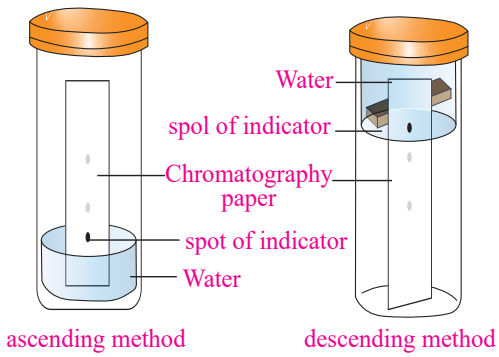


Figure 15.9 Ascending and descending paper chromotography

15.4.2 Locating agents 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.

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 locate the spots. 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.

15.4.3 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 15.10** the chromatogram shows that the sample A contains both components A and B. The R_f values for B and C are given by:

$$R_f\,(A) = \frac{x}{z} \qquad R_f\,(B) = \frac{y}{z}$$

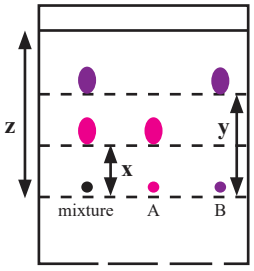


Figure 15.10 Chromatogram

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. 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.

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.

15.4.4 Applications of Paper Chromatography

i. The techniques of chromatography are very useful in organic synthesis for separation, isolation and purification of the products. They are equally important in qualitative and quantitative analyses and for determination of the purity of a substance.

ii. 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?

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

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

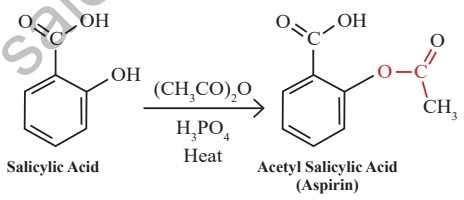
15.5 HOW TO CHECK THE PURITY OF THE PRODUCT?

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.

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 mixture is then filtered to get aspirin as a crude product. It is then crystallized in water as polar solvent.

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, its melting point is determined. If the product melts sharply, in this case at exactly 136°C then the product is considered as pure. If the product shows a melting point which is not sharp rather it melts within a range of temperature, it is considered as an impure product.



Salicylic Acid — Acetyl Salicylic Acid (Aspirin)

ii. Another way of assessing 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 the R_f value.

Quick Check 15.5

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

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

EXERCISE

MULTIPLE CHOICE QUESTIONS

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

II. The comparative rates at which the solutes move in paper chromatography depend on:

a) 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 phases
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

SHORT ANSWER QUESTIONS

Q.2 Attempt the following short-answer questions:

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

b. What is the function of fluted filter paper?

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

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

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

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

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

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

i. What is the basic principle of paper chromatography?

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

k. Differentiate between adsorption and partition chromatography.

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

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

DESCRIPTIVE QUESTIONS

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

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

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