

1. Mixtures and Separation Techniques

1.1 Introduction

There are more than one hundred **elements** from which all substances are â€˜builtâ€™. Chemistry is the study of these â€˜building blocksâ€™ and the **compounds** they form. But the compounds formed by these elements are usually mixed up in nature, ie they exist as **mixtures**.

Common examples of mixtures of compounds found in nature are:

- (i) sodium chloride dissolved in water in the salt wells of Uburu in Abia State,
- (ii) tin oxide which is mixed with sandy and rocky impurities in mines of the Jos plateau,
- (iii) petrol mixed with other hydrocarbons like diesel, and kerosine in the petroleum obtained from our creeks,
- (iv) sucrose mixed with water in cane sugar, and
- (v) Oxygen gas which is mixed with nitrogen, carbon dioxide, etc in air.



Plate 1.1(a) A salt well



Plate 1.1 (b) Tin ore and the tin mines in Jos



Plate 1.1 (c) Petrol and oil gas wells in Port Harcourt

To obtain these compounds for study as well as for our own use, we have to separate them from the other components. The separation of the components of these mixtures to obtain the compounds we want, is an important function of chemists.

Before we start learning the techniques for separating mixtures to obtain pure compounds, let us first find out how we can identify pure compounds.

1.2 Test of purity of a substance

Certain properties of chemical substances change when there are impurities in them, but remain constant if the substances are pure. Two of such properties are often used to characterise substances.

These are their melting (or freezing) points, and their boiling points. The melting and boiling points of some pure substances are given in Table 1.1.

TABLE 1.1 Melting and boiling points of some pure substances

Substance	Melting Point ($^{\circ}\text{C}$)	Boiling Point ($^{\circ}\text{C}$) (at atmospheric pressure)
Water	0	100
Ethanol	-117	78
Benzene	5.5	80
Iron	1535	3000
Sodium	98	890
Sulphur	115	445
Naphthalene	80.2	218
Sodium chloride	801	1467
Propanone (Acetone)	-95.4	56

Experiment 1.1

To Investigate the Effect of Dissolved Solute on the Melting Point of a Substance.

Grind a half spatula-full of naphthalene in a mortar with pestle. Pack some of the powdered naphthalene into a melting point capillary tube sealed at one end to a height of about 1mm. Secure the melting point tube to a thermometer with a thin rubber band. Immerse the thermometer into paraffin oil contained in a small beaker, making sure that the bulb of the thermometer does not touch the sides or bottom of the beaker (Figure 1.1). Heat the paraffin oil with a low bunsen flame, stirring regularly.

When the naphthalene crystals begin to show signs of crumbling, note the temperature and remove the flame so that the mercury inside the thermometer rises very slowly. Read the temperature at which the last crystal of naphthalene crumbles and the solid turns to liquid. The range of temperature through which the solid melts is very narrow. Usually, if the solid starts to melt at 79.5°C , the whole mass will melt completely before the temperature reaches 80.5°C . The melting point (m.p.) is described as sharp and the mid-point of this range (80°C) is the melting point of naphthalene.

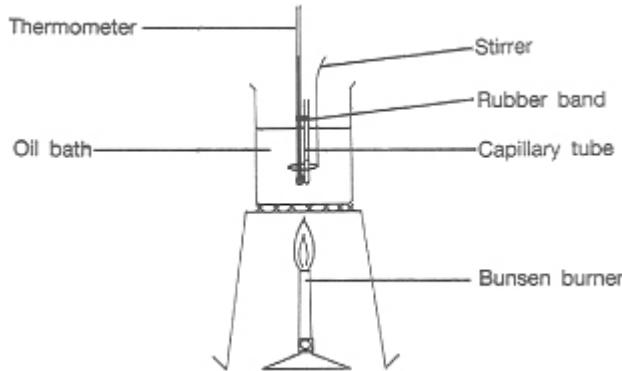


Figure 1.1 Melting point determination

Next grind a mixture of nine parts of naphthalene and one part of camphor together, and pack some of this into a melting point tube to a height of about 2mm. Repeat the melting point determination.

While the pure naphthalene melts at 80°C , the mixture of naphthalene and camphor will melt at a lower temperature, showing that **impurities lower the melting points of substances**. The melting point is not sharp in this case as its range is usually more than 2°C . This means that a sample of naphthalene whose melting point is found to be 80°C must be pure.

Experiment 1.2 To Investigate the Effect of Dissolved Solute on the Boiling Point of a Liquid

Put about 5cm^3 of distilled water into a boiling tube immersed in a 100cm^3 beaker containing paraffin oil as shown in Figure 1.2. Insert a thermometer and stirrer into the boiling tube.

Heat the oil bath with a small bunsen flame. Continue heating, watching the rising level of mercury in the thermometer. Record the steady temperature when the mercury level no longer rises even though the oil bath is still being heated. This is the boiling point of water at that atmospheric pressure.

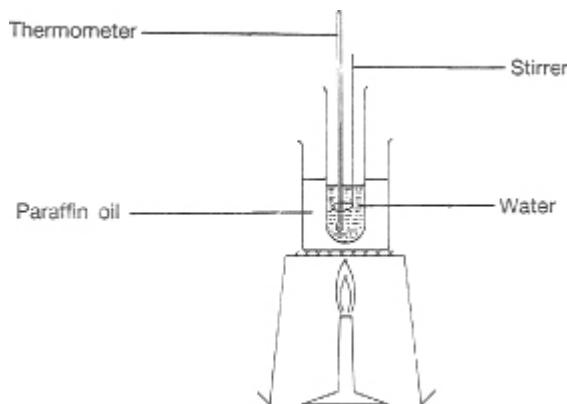


Figure 1.2 Boiling point determination

Add a spatula-full of sodium chloride to 5cm^3 of distilled water and

repeat the experiment. You will find that while pure water boils at about 100°C , the solution of sodium chloride in water boils at a higher temperature. **Dissolved impurities raise the boiling points of liquids.**

1.3 Physical and chemical changes

The changes which matter undergo under different conditions can be classified into two broad areas – physical (or temporary) changes, and chemical (or permanent) changes. No new substances are formed during a physical change. Physical changes are easily reversed by physical means such as evaporation, cooling and condensation. In chemical changes new substances are formed. Chemical changes are not readily reversed.

Since no new substances are formed during a physical change, there is usually no gain or loss in mass as in the melting and solidification of candles. Heat change is limited to that due to latent heat effects. In a chemical change, however, there may be a gain or loss in the mass of the products, compared to the original substances, and heats of reaction usually accompany the change. Some changes have been classified as physical or chemical in Table 1.2.

TABLE 1.2 Physical and chemical changes

	Change	Physical	Chemical
1.	Sugar dissolves in water	✓	“
2.	A match stick burns in air	“	✓
3.	Rusting of iron	“	✓
4.	Liquids freeze to solids	✓	“
5.	Evaporation of liquids	✓	“
6.	Zinc effervesces in dilute HCl	“	✓
7.	Palm wine goes stale overnight	“	✓
8.	Stain is removed from fabric with a solvent	✓	“

Some physical processes will be employed to separate mixtures in the following section.

1.4 Separation of mixtures

A mixture contains two or more substances that are not chemically bound together. Techniques used to separate components of mixtures include decantation, filtration, evaporation, distillation, crystallization, precipitation, sublimation and chromatography. The choice of a separation technique depends on the properties of the mixture. Let us consider these techniques one by one.

1.5 Filtration

If one component of a mixture is insoluble in a given solvent while the others are soluble, then the insoluble component can be separated from the soluble ones by shaking the mixture in the solvent, and decanting off the solution, or filtering it.

Experiment 1.3 To Separate a Mixture of Sand and Common Salt (Sodium Chloride)

Put the mixture of sand and salt into a beaker and add some water to it. Stir the mixture in water. If all the salt does not dissolve, that is if some white solids remain, add more water and stir. Fold a filter paper into a cone and fit this into a funnel. Pour the liquid portion of the mixture into the filter paper and collect the **filtrate** in a conical flask, arranged as shown in Figure 1.3. The filter paper should never be more than two-thirds full.

Most of the sand will be left in the beaker when all the solution of the salt in water has passed through the filter paper into the conical flask. Add a little more water to the beaker, stir, and pour the liquid into the filter paper, still leaving the bulk of the sand in the beaker. This process is called filtration by decantation. Remove the flask containing the filtrate and replace with another conical flask, before transferring the sand from the beaker into the filter paper, with a spatula. Allow the sand to drain dry.

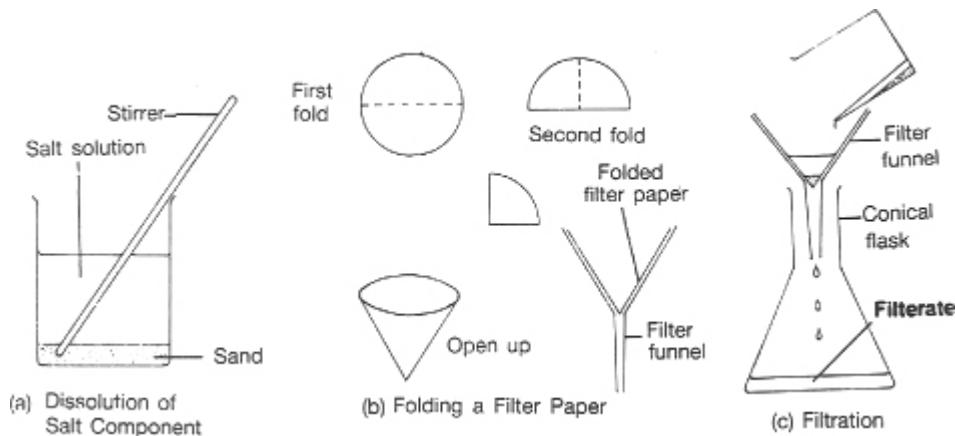


Figure 1.3 Filtration process

1.6 Evaporation

Dissolved solids can be separated from liquid solvents by evaporation. This is carried out in an evaporating basin. The solvent molecules escape either when the solution is heated, or when it is just left to stand, leaving the less volatile solute particles behind.

Experiment 1.4 Evaporation of the Filtrate from Experiment 1.3

Put the filtrate from Experiment 1.3 into an evaporating basin. Heat the basin on a wire gauze with a bunsen flame (Figure 1.4). Continue the heating until all the solvent has evaporated. A white crystalline solid of the salt (sodium chloride) is left.

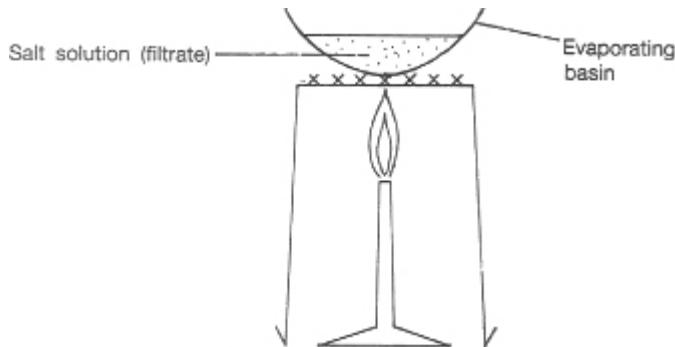


Figure 1.4 Evaporation

1.7 Distillation

Distillation is employed when a liquid solvent is to be recovered. The solution to be distilled may be a mixture of a solid solute in a liquid solvent, or of two or more liquids, in solution. To separate a solid solute in liquid solvent simple distillation is used. But when two or more liquids are involved, they must have widely different boiling points for simple distillation to be applied. If their boiling points are not widely separated, fractional distillation should be used.

Experiment 1.5 Separation of A Mixture of Ethoxyethane (Diethyl Ether) and Water by Simple Distillation

The boiling points of ethoxyethane (45°C), and water (100°C), are widely different. Separation of a mixture of the two liquids can therefore be effected by simple distillation.

Put the mixture of the two liquids into a distillation flask. Connect a thermometer and a Liebig's condenser to the flask as shown in Figure 1.5. Heat the distillation flask with a bunsen flame, while passing cold water through the condenser.

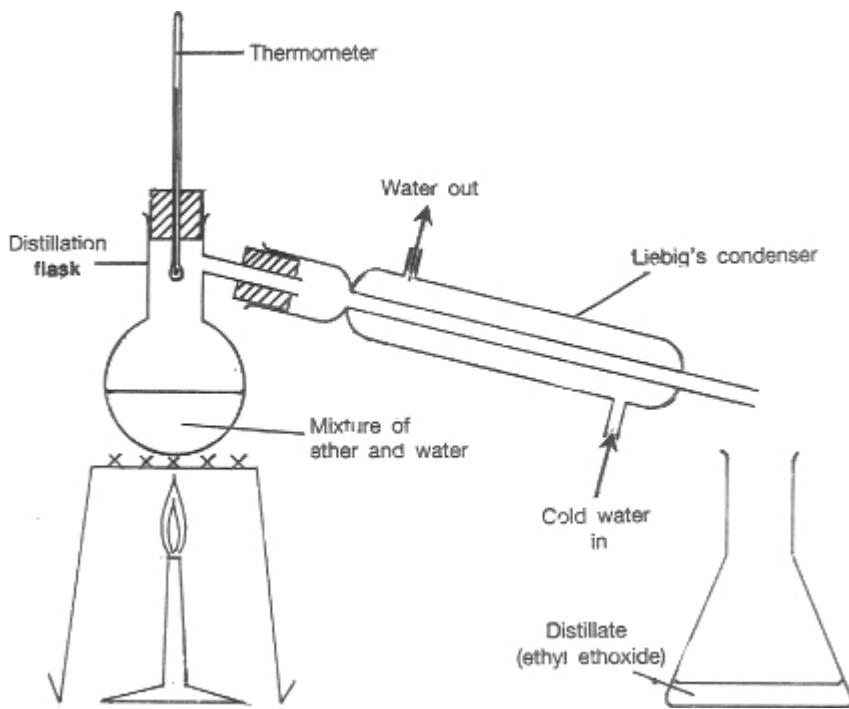


Figure 1.5 Simple distillation

Caution: the receiver must be kept far from the bunsen flame as Ethoxyethane is highly inflammable, if possible use a hot plate or heating mantle in place of a bunsen flame.

Collect the distillate coming off at 45°C . When the temperature rises up to 50°C remove the source of heat till the temperature falls back to 45°C . When no more **distillate** comes over, stop heating. The distillate is pure ethoxyethane. The liquid remaining in the distillation flask is not likely to be pure water because some ether vapour invariably condense in the flask and return to the bulk of the liquid.

Mixtures of liquids whose boiling points are close such as petrol and kerosene, are separated by **fractional distillation**. On heating the mixture, vapours of both liquids come up. They are sent into a **fractionating column**. There, vapours of the higher-boiling component condense (turn back to liquid) and return to the flask where heating is taking place. Only the vapours of the lower-boiling component get to the condenser where they condense on cooling, and are collected as distillate.

This process is used in the refining of petroleum, during which petrol, kerosene, diesel oil and lubricating oils condense at different temperatures along the fractionating column. This is discussed in Chapter 9.

Experiment 1.6 Separation of Petrol and Kerosene By Fractional Distillation
caution: Put out all flames in the vicinity

Put the mixture into a distillation flask. Connect a fractionating column, a thermometer, a condenser and a receiver as shown in Figure 1.6. Add some anti-bumping agent (e.g. glass beads or porous pot chips) to ensure smooth boiling.

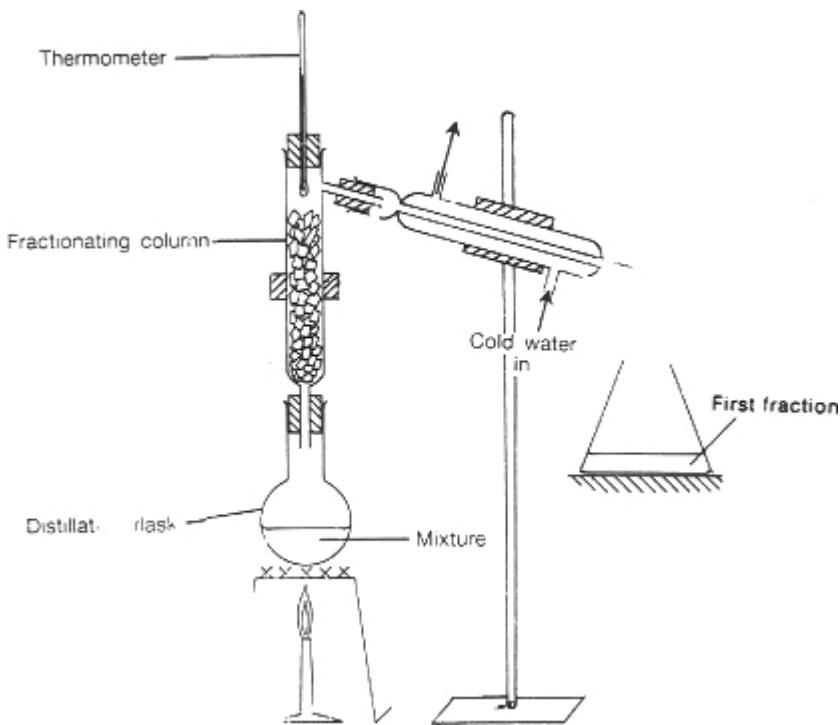


Figure 1.6 Fractional distillation

Heat the flask with a hot plate or heating coil until vapours start to condense in the condenser. The reading of the thermometer should then be about $60\text{ }^\circ\text{C}$. Collect the distillate coming over between this temperature and $90\text{ }^\circ\text{C}$. When no more distillate comes over, replace the receiver and raise the temperature to about $110\text{ }^\circ\text{C}$. Vapours start to condense again at this temperature. Collect distillates coming over up to a temperature of $140\text{ }^\circ\text{C}$.

Determine the boiling points of the two fractions. The first fraction is petrol (CARE!). The second is kerosene.

Exercise 1A

How best can you separate a mixture of ethanol (boiling point $78\text{ }^\circ\text{C}$), and water (boiling point $100\text{ }^\circ\text{C}$)?

1.8 Sublimation

When a solid mixture contains a component that **sublimes**, (i.e. changes into the vapour state from the solid state without first becoming liquid), then separation can be effected by sublimation. This involves heating the solid mixture and collecting the vapour of the

component that sublimes.

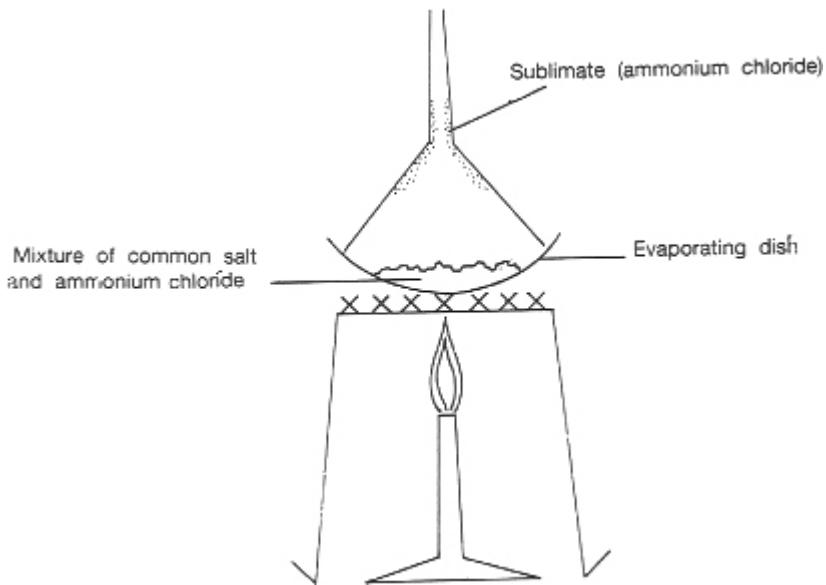


Figure 1.7 Separation by sublimation

The vapour resolidifies on a cold surface above the heated container.

Only a few substances sublime. They include ammonium chloride, iodine, sulphur and naphthalene. This method of separation is therefore of limited application.

Experiment 1.7 To Separate a Mixture of Ammonium Chloride and Common Salt by Sublimation

Put the mixture into an evaporating dish. Place a glass funnel over the dish, then heat it. White fumes will rise from the evaporating dish and condense a white deposit of ammonium chloride in the inside of the funnel (Figure 1.7).

1.9 Crystallization

Crystallization is a process used to obtain a solid from its solution in a liquid. It is preferred to evaporation when the crystals of the solid need some water of crystallisation to form well or if a high temperature will change its composition. Such a solution is first concentrated by evaporation, before being cooled. Crystals separate out of the concentrated solution, and are collected by filtration.

Experiment 1.8 Crystallisation of Copper (II) Tetraoxosulphate (VI) Pentahydrate from Aqueous Solution

You are provided with a dilute solution of copper(II) tetraoxosulphate(VI) in water. Heat the solution in an evaporating dish to concentrate it. While heating, occasionally withdraw a sample of it with a dropper pipette, into a test-tube, and cool this under running

tap water to see if crystals form. This is testing for saturation.

When the solution is sufficiently concentrated, crystals will form inside the test-tube on cooling. When this occurs, stop heating and leave the solution in a safe place to cool. After sometime, blue crystals of the salt may be observed growing in the solution. When the solution has cooled completely and no more crystals grow, filter off the crystals through a filter paper, and rinse it with cold water from a wash bottle. Dry it between fresh filter papers. The process is illustrated in Figure 1.8.

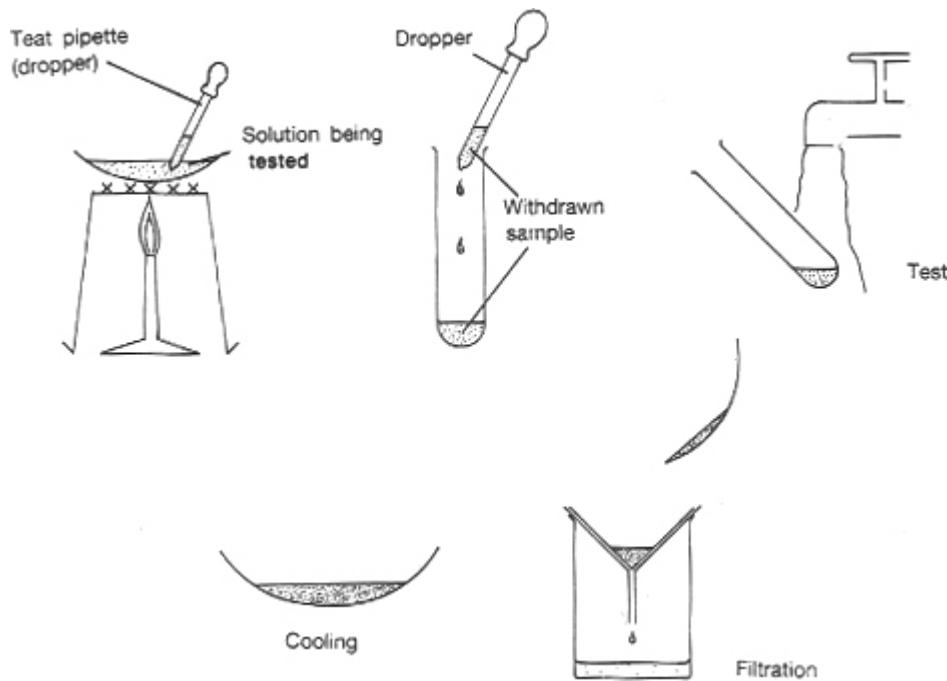


Figure 1.8 Processes in crystallization

Fractional crystallization

If the solubilities of two solids in one liquid solvent are widely different, then the solids can be separated by fractional crystallization. The mixture is dissolved in a common solvent, and the solution is concentrated by heating to evaporate the solvent. We test for saturation as in Experiment 1.8. We stop heating and allow the solution to cool when it is saturated. The component with the lower solubility crystallizes out first and is filtered off. The filtrate is further concentrated by evaporation. When it is saturated, it is left to cool. The cooling results in the crystallization of the component with the higher solubility.

Experiment 1.9 To Separate a Mixture of Potassium Trioxochlorate(V) and Potassium Heptaoxodichromate(VI) by Fraction Crystallization

Put about 5g of the mixture containing equal amounts of the two salts into an evaporating dish. Add about 15cm³ of water and heat to

dissolve. Stir while heating. If any solid remains while the liquid is boiling, add more water, but just enough to get a saturated solution. When all the solid has dissolved, cool the solution in cold water, and allow crystals to form.

Decant the liquid into a clean beaker. Redissolve the crystals with little fresh water and boil, with stirring. Cool, to form crystals. This process of redissolving the crystals, heating and cooling to crystallize is called **recrystallisation**. It purifies the less soluble component by ensuring that part of the more soluble component which crystallized along with it is redissolved. Filter off the crystals. Wash with little cold water and dry on filter paper in a funnel. The white crystals are crystals of potassium trioxochlorate(V).

Combine the filtrate from the recrystallization process with that from the first decantation. Concentrate the combined solutions by evaporation, testing occasionally for saturation. If any more white crystals form; collect these by filtration or decantation, then concentrate the remaining solution further till orange crystals of potassium heptaoxodichromate(VI) form. Cool, and filter off the crystals. Dry in an oven.

Exercise 1B

On what property of the two salts does this technique for their separation depend?

1.10 Precipitation

Precipitation involves throwing a solid solute out of solution. Its application in separation of mixtures makes use of the difference in solubilities of a solid in two miscible liquids. The solid, dissolved in one of the two liquids in which it is more soluble, is precipitated (forced out of solution) by the addition of the second liquid in which it is less soluble.

Experiment 1.10 Precipitation of iron(II) tetraoxosulphate(VI) In Water
Prepare a solution of Iron(II) tetraoxosulphate(VI) in water by dissolving about 5g, of the salt. To recover the salt we use the process of precipitation by adding an equal volume of ethanol to the solution (Figure 1.9). Ethanol and water are miscible, but iron(II) tetraoxosulphate(VI) is insoluble in ethanol.

The pale-green solid precipitates out of solution. Filter it off and dry in the oven.

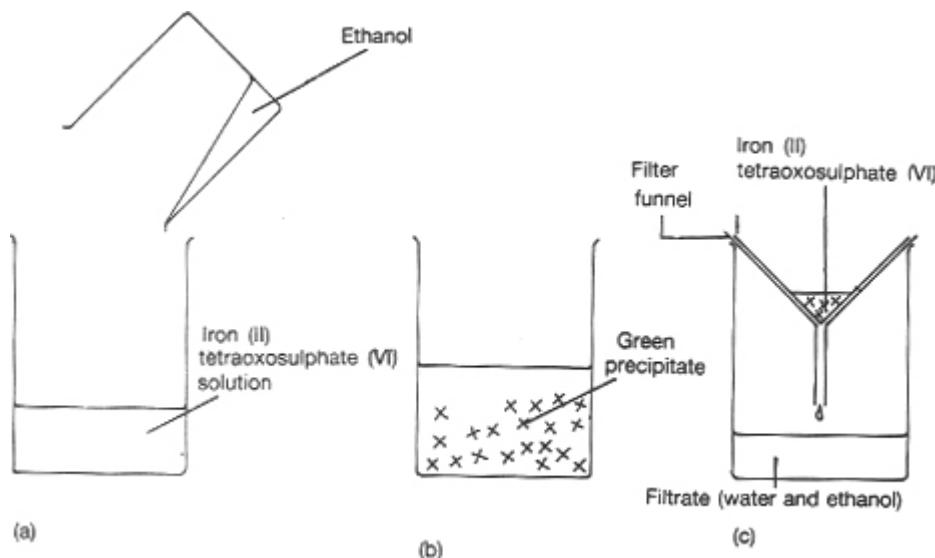


Figure 1.9 Precipitation of iron(II) tetraoxosulphate(VI) from aqueous solution

1.11 Chromatography

Separation by chromatography makes use of the different rates of movement of the constituents in a mixture, over an adsorbent medium. The adsorbent medium may be a sheet of adsorbent paper or a column packed with alumina, silica or other such materials.

Chromatography is suitable for the separation of components in a mixture, which are present in very small amounts. Many variations of this technique are presently used in the field of analytical chemistry. The separated constituents are identified by various means, the commonest being by development into different coloured bands. Each coloured band is composed of a particular chemical compound.

Experiment 1.11 To Separate the Constituents of Chlorophyll by Paper Chromatography

Cut some grass from the field and leave to dry for some time in the sun. Put these into a mortar and crush into a mash with a pestle. Add a little propanone (acetone) to dissolve out the chlorophyll, while stirring with the pestle. Concentrate the solution by evaporation.

Cut a length of filter paper, 1cm wide and 15-20cm long. Draw a line with a pencil across the width, about 3cm from one end of the paper. Make a mark at the centre of the line.

Using a dropper pipette, place a drop of the chlorophyll solution at the point marked on the line. Allow the drop to dry, then spot a second drop at the same position. Leave this to dry also.

Put about 5cm^3 of propanone into a wide boiling tube. Hang the paper with a hook onto a cork and place it into the tube with the end where the drop of chlorophyll solution was spotted dipping into the

propanone. The level of propanone in the tube should be below the line marked on the paper. Fit the cork tightly to the boiling tube as in Figure 1.10., and leave for several hours.

The propanone rises up the paper by capillary action. As it does so, it carries the components of the chlorophyll solution along. When the solvent front is near the other end of the paper, remove the paper from the tube. Allow it to dry in air. Different coloured bands will be seen on the paper. Each band represents a component of chlorophyll. The different components are carried up the paper at different rates, thus effecting separation.

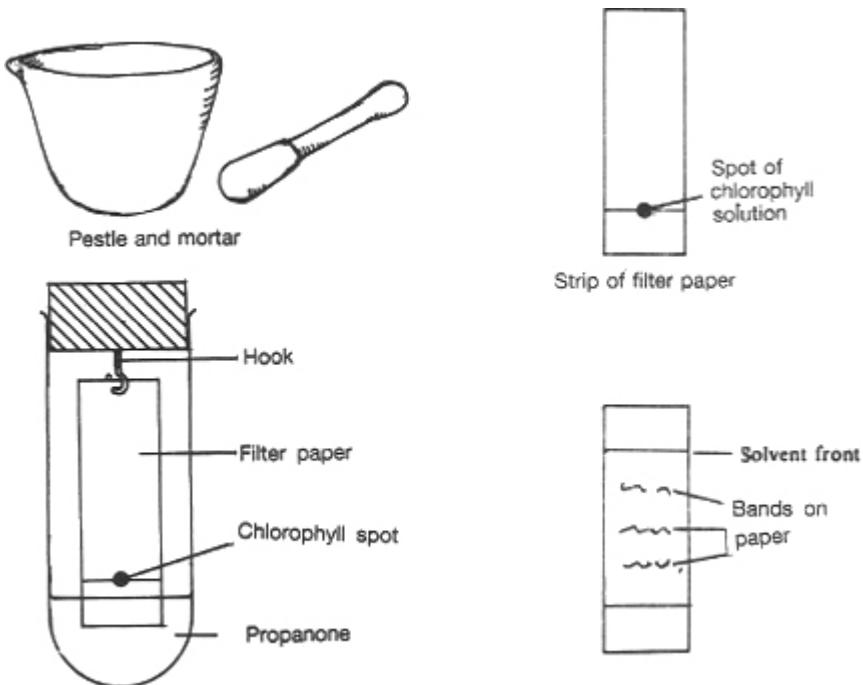


Figure 1.10 Paper chromatography of chlorophyll

Colourless components of a mixture can equally be separated by chromatography. There should however be a means of identifying them after separation. One method of doing this is to spray the paper after separation, with a suitable dye which reacts with the components to form coloured derivatives. Another method is to observe the paper with ultraviolet light if the components possess fluorescent properties.

Experiment 1.12 Separation of the Cations of Nickel, Cobalt and Zinc by Paper Chromatography

A mixture containing about equal amounts of nickel tetraoxosulphate(VI), cobalt(III) chloride and zinc tetraoxosulphate(VI) is provided. Dissolve a very small amount of this mixture in enough water to make a saturated solution. Cut a strip of filter paper about 20-25cm long and 3cm wide. A longer strip of paper is needed than for the last experiment. Draw a thin line with pencil, about 1cm, from one end of the strip of paper. Spot a drop of the solution at the centre of

this line. Allow it to dry, then spot another drop at the same point. Dry this also.

Prepare a mixture of propanone, concentrated hydrogen chloride acid (hydrochloric acid) and water in the ratio 43.5:4.0:2.5 cm³ by measuring the required volumes with a burette for accuracy. Pour some of this into a measuring cylinder to such a level that the spotted edge of the strip of paper dips into it, while hanging from the mouth of the cylinder as shown in Figure 1.11.

Allow the set up to stand until the solvent front has moved to near the other end of the strip of paper. Remove the paper and dry it in air, then spray it with ammonia to neutralise the acid. Again allow to dry, and finally spray it with ammonium sulphide solution, or expose it to hydrogen sulphide gas from a source such as the **Kippâ€™s apparatus**, in a fume chamber.

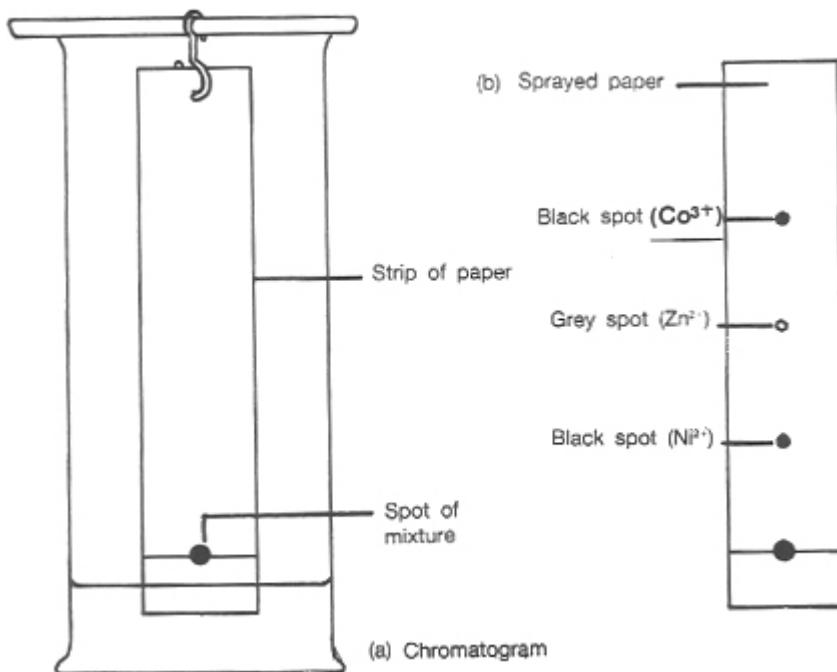


Figure 1.11 Paper Chromatography of Co^{3+} , Zn^{2+} and Ni^{2+}

A black spot near the original spot represents nickel ion (Ni^{2+}). Another black spot near the top of the paper represents cobalt ion (Co^{3+}) while the grey **spot in-between is due to zinc ion (Zn^{2+})**

Exercise 1c:

Why would you consider each of the separation methods a physical rather than a chemical process?

Worked example:

A chemist prepares a compound in his laboratory and finds it to melt between 178-182°C. What can you say about the purity of this compound?

Solution:

The compound is impure. Pure substances have sharp melting points, that is, they melt within a small range of temperature. This compound would have **been considered pure if it melted, say between 178-179°C.**

Chapter Summary

1. Substances are rarely found free and pure in nature. Chemists often have to isolate and purify them.
2. Various separation techniques are employed in the purification. The major techniques include filtration, distillation, crystallization, precipitation, and chromatography.
3. The test of purity is either a sharp melting point which is constant for a given solid substance, or a boiling point which is also constant at atmospheric pressure for a given liquid.
4. The following terms used in the unit should be understood and noted: solution, solvent, solute, concentrated, dilute, saturated, distillation, distillate, chromatogram, sublimate, filtrate, melting point and boiling point.

Assessment

1. Name the separation techniques you would use to separate the following mixtures:
 - (a) Ethanol (b.p 78°C) and benzene (b.p 80°C);
 - (b) Iodine crystals and iron filings;
 - (c) Silver chloride and sodium chloride;
 - (d) Methanol (b.p 65°C) and water (b.p 100°C);
 - (e) Ink.
2. Define the terms “distillate”, “sublimate” and “filtrate”.
3. How can you show that the products of heating 5.6g of iron and 3.2g of sulphur is a compound, and not a mixture?
4. What properties of the components of a mixture are employed in their separation in:
 - (a) distillation;
 - (b) crystallization;
 - (c) sublimation;
 - (d) chromatography?
5. A drop of a liquid extract from a plant is spotted on a thin strip of paper and dried. The end of the paper with the spot is then immersed in propanone for two hours. At the end of this period the paper is again dried, then sprayed with an organic solvent. Three

spots appear at different points on the paper.

- (a) How many components are present in the extract?
 - (b) What is the name of this separation technique?
 - (c) What property of the components enables their separation?
6. A chemist isolated a mixture from a West African medicinal plant and ran two paper chromatograms of it, one in ethanol and the other in benzene. Benzene gave two spots on the paper and ethanol four. Which of the two solvents will be more suitable for purifying the components of the mixture?
- (WAEC)
7. (a) Give four differences between mixtures and compounds.
(b) If you are provided with (i) a mixture of iron fillings and sulphur, and (ii) a compound of iron and sulphur, how would you demonstrate that (i) is a mixture, and (ii) is a compound?
(c) Explain why an aqueous solution of sodium chloride is a mixture of sodium chloride and water.
- (WAEC)
8. What method can be used to separate the chloropentane formed in a reaction between chlorine and excess pentane from the excess pentane in the reaction mixture?