CHAPTER 2

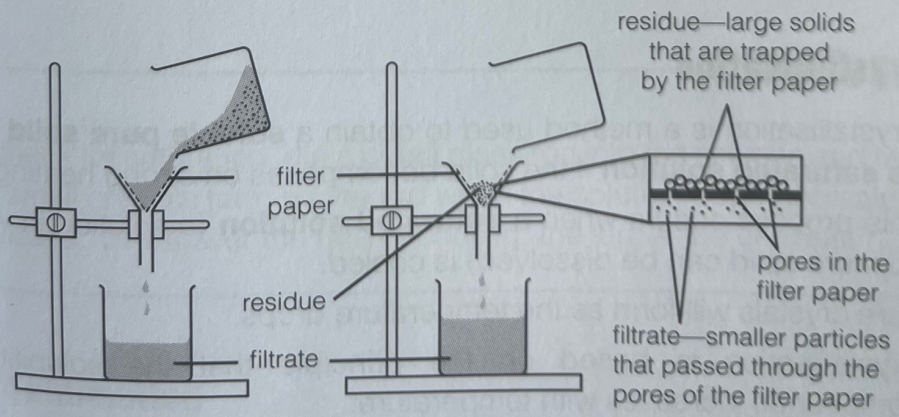
Methods of Purification and Analysis

NOTES

1. **Purification** is a process used to separate pure substances from mixtures or from impurities.
2. There are various methods of purification and each method is used to separate substances from different kinds of mixtures.

2.1 Filtration

1. Filtration is a method used to separate **insoluble solids from a liquid-solid mixture**.
2. Large particles of the solid will be trapped by the pores of the filter paper while the smaller liquid particles pass through.



Take Note!

There are soluble salts such as silver nitrate and copper(II) sulfate and insoluble salts such as calcium carbonate and barium sulfate. See Chapter 8 for the table of soluble and insoluble salts.

2.2 Evaporation to Dryness

1. Evaporation to dryness is a method used to obtain a **soluble solid from its solution**.
2. The solution is heated until all the water has evaporated.
3. The soluble solid must be stable to heat so that it does not decompose on heating and must not contain water of crystallisation.

Take Note!

Water of crystallisation refers to the water molecules that make up the structure of a salt crystal. Hydrated copper(II) sulfate ($CuSO_4.5H_2O$) is an example of a substance that contains water of crystallisation.

2.3 Crystallisation

1. Crystallisation is a method used to obtain a **soluble pure solid from its saturated solution** if the solid decomposes on strong heating.
2. This process occurs when a **saturated solution** (a solution in which no more solid can be dissolved) is cooled.
3. Pure crystals will form as the temperature drops.
4. Crystallisation is based on the principle that the solubility of compounds increases with temperature.

Steps to obtain crystals:

1. Dissolve the impure solid in water until no more of it can be dissolved.
2. Filter the solution to remove the extra undissolved solid and insoluble impurity.
3. Heat the solution till saturation.
4. Leave the hot saturated solution to cool. Crystals will start to form in the solution.
5. The cooled solution is then filtered to obtain the pure crystals. The crystals are dried by pressing them gently between sheets of filter paper.



Take Note!

A crystal is pure because only the particles of the compound arrange together to form a highly ordered structure. Impurities are left behind in the solution.

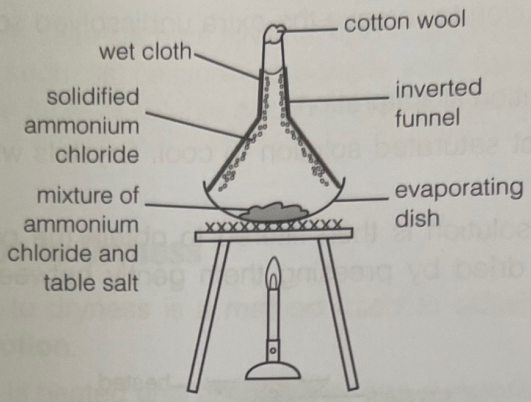
Practical Tips

To check for saturation, dip a clean glass rod into the solution and remove it. If small crystals form on the rod when the solution cools, the solution is saturated. The slower the rate of cooling, the larger the crystals.

2.4 Sublimation

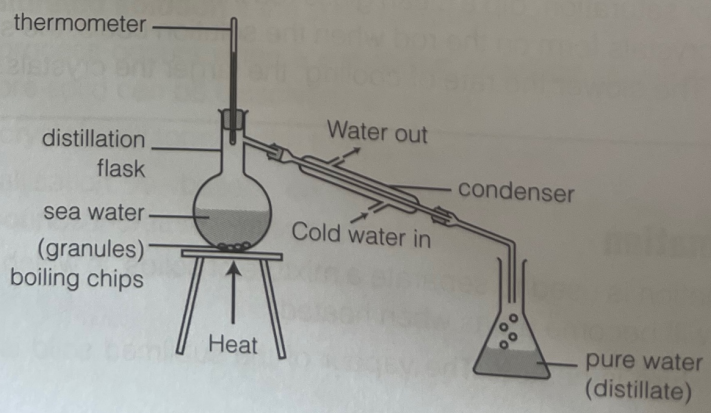
1. Sublimation is used to separate a mixture of solids, in which one of the solids will become a gas when heated.
2. The mixture is heated. The vapour of the sublimed solid is cooled to yield the solid.

3. Solids that sublime on heating include ammonium chloride, iodine and carbon dioxide (dry ice).



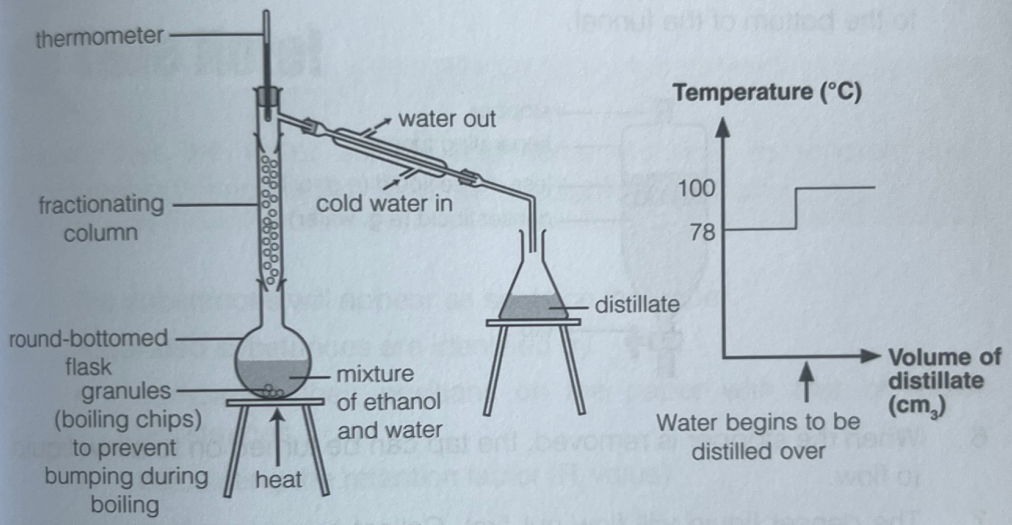
2.5 Simple Distillation

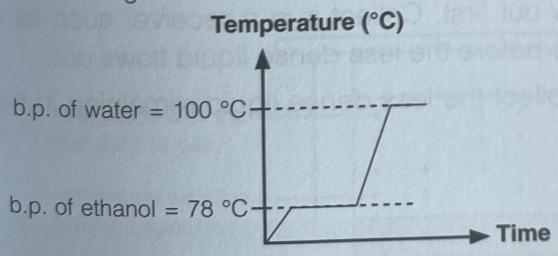
1. Simple distillation is used to separate **a pure solvent (liquid) from a solution**.
2. The solution is heated in a distillation flask connected to a **condenser**.
3. The liquid boils and its vapour enters the cold condenser where condensation takes place.
4. The vapour is converted back into its pure liquid form and collected as the **distillate**.
5. For example, pure water can be obtained by carrying out simple distillation on sea water.



2.6 Fractional Distillation

1. This method is used to separate **a pure liquid from a solution of two or more miscible liquids** with different boiling points.
2. **Miscible liquids** are those that are completely soluble in one another when mixed.
3. The liquids are separated according to their **boiling points**.
4. The liquid with the lowest boiling point will be distilled first.



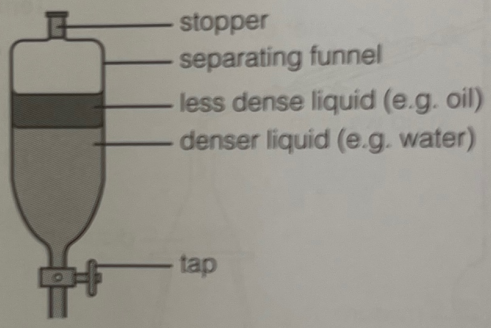


Take Note!

1. Principles are concepts. Take note of the different principles behind each method of separation. For example, fraction distillation is based on the principle that the liquid with the lowest boiling point distills off first.
2. Organic liquids and water do not usually mix. Ethanol and ethanoic acid are few of the organic liquids that mix with water well.

2.6 Using a separating funnel

1. A separating funnel is used to separate **immiscible liquids**.
2. Immiscible liquids are liquids that do not mix with one another.
3. separating funnel has a stopper covering the top and a tap at the bottom.
4. A mixture of two immiscible liquids will form two separate layers.
5. The less dense liquid will be on top of the denser liquid which will sink to the bottom of the funnel.



6. When the stopper is removed, the tap can be turned on to allow liquid to flow.
7. The denser liquid will flow out first. Collect it in a receiver such as a beaker and turn off the tap before the less dense liquid flows out.
8. Use another receiver to collect the less dense liquid remaining in the separating funnel.

Take Note!

Most organic compounds such as fossil fuels and vegetable oil are insoluble in water and will float on water.

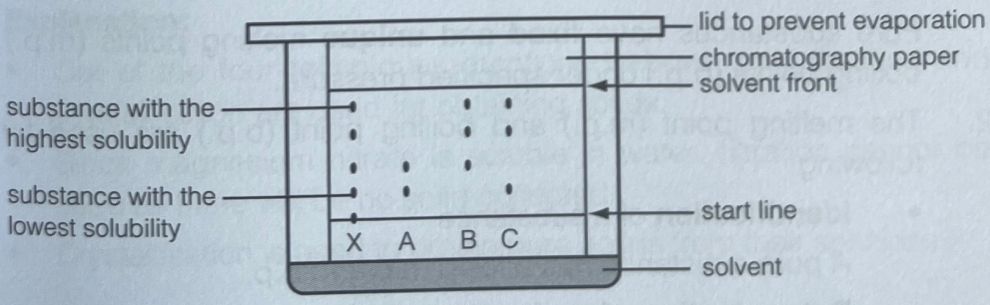
2.7 Paper chromatography

1. Paper chromatography is used in the separation and identification of both coloured and colourless mixtures such as drugs and dyes.
2. The solvent comes into contact with the bottom of the chromatography paper and moves up by capillary action.
3. Different substances in the mixture become separated as they travel at different rates due to their **varying solubilities in the solvent**.

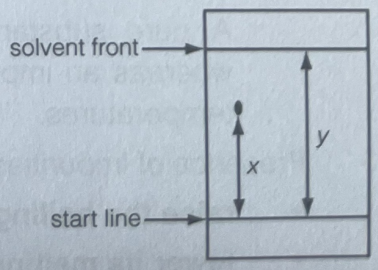
Take Note!

Some inks are water soluble and some are not. To separate the components of an ink that is not water soluble, use ethanol.

4. The substances will appear as spots on the paper.
5. Separated substances are identified by
   - comparing their positions on the paper with that of known substances,
   - calculating the retention factor (R_f value)



$$R_f = \frac{x}{y} = \frac{\text{distance travelled by the solute up the paper}}{\text{distance travelled by the solvent}}$$



6. For substances that share the same R_f value, chromatography can be performed with a different solvent to distinguish them.
7. For separating **colourless** compounds, **locating agents** are used to show the position of these compounds in the chromatogram.
8. Locating agents are substances that react to form coloured products with the colourless compound.

Practical Tips

1. Do not allow the spot to spread while applying the spot. Allow the drop applied to dry before placing the paper into the solvent. Make sure that the drop is above the solvent when the chromatography is dripped into the solvent.
2. Draw the starting line with a pencil, not with ink which may dissolve in the solvent and interfere with the results.
3. Make sure the starting line is above the solvent, otherwisde, the spot will dissolve in the solvent.

2.8 Analysis of Purity of Substances

1. Pure substances have **fixed** and **unique** melting points (m.p.) and boiling points (b.p.) under specified pressure.
2. The melting point (m.p.) and boiling point (b.p.) are used for the following:
   - **Identification** of a substance

     A pure substance has a fixed m.p and b.p.
   - **Determination of purity** of a substance

     A pure substance melts and boils at specific temperatures whereas an impure substance melts and boils over a range of temperatures.

3 Presence of impurities will:
   - **raise the boiling point** of a substance, and
   - **lower its melting point**.

4. Purity is useful in many ways in daily life. Its analysis can be used to:
   - Check for approved dyes in food
   - Check for drug abuse in sports by testing the urine of athletes
   - Ensure purity of active ingredients in drugs
   - Check for pesticides and herbicides in vegetables

Worked Examples

1. Solid magnesium is reacted with aqueous nitric acid to form a solution of magnesium nitrate. Which of the following techniques is the most suitable for obtaining solid magnesium nitrate from its solution?

   A Filtration
   B Using a separating funnel
   C Crystallisation
   D Simple distillation

   Explanation:
   - Out of the four techniques mentioned above, only filtration and crystallisation are used for obtaining solids.
   - Since magnesium nitrate is soluble in water, filtration cannot be used as there will be no solid collected.
   - Crystallisation is used to obtain pure solids from their solutions.

   Answer: C

2. Which of the following is a pure compound?

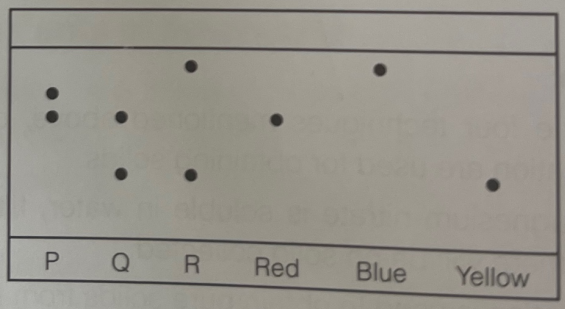
A A greenish solution
B A colourless liquid that boils at 70°C
C A brown solid that melts between 109°C and 113°C
D A white residue obtained from filtration of a mixture

Explanation:

- Purity of a substance can be identified by its melting and boiling points.
- A pure substance has fixed and unique melting and boiling points.
- Impure substances melt and boil over a range of temperatures.

Answer: B

3. The diagram below shows a chromatogram of three unknown substances, P, Q and R, and three coloured dyes.



(a) Which dye(s) is/are present in each of the unknown substances P, Q and R? [3]

(b) Which substance contains an unknown dye? [1]

(c) Paper chromatography was performed again with a new unknown substance, S. It was observed that S shows the same results as P. What can be done to differentiate P and S? [1]

Solution:

(a) P contains red dye. [1]

Q contains red and yellow dyes. [1]

R contains yellow and blue dyes. [1]

(b) Substance P [1]

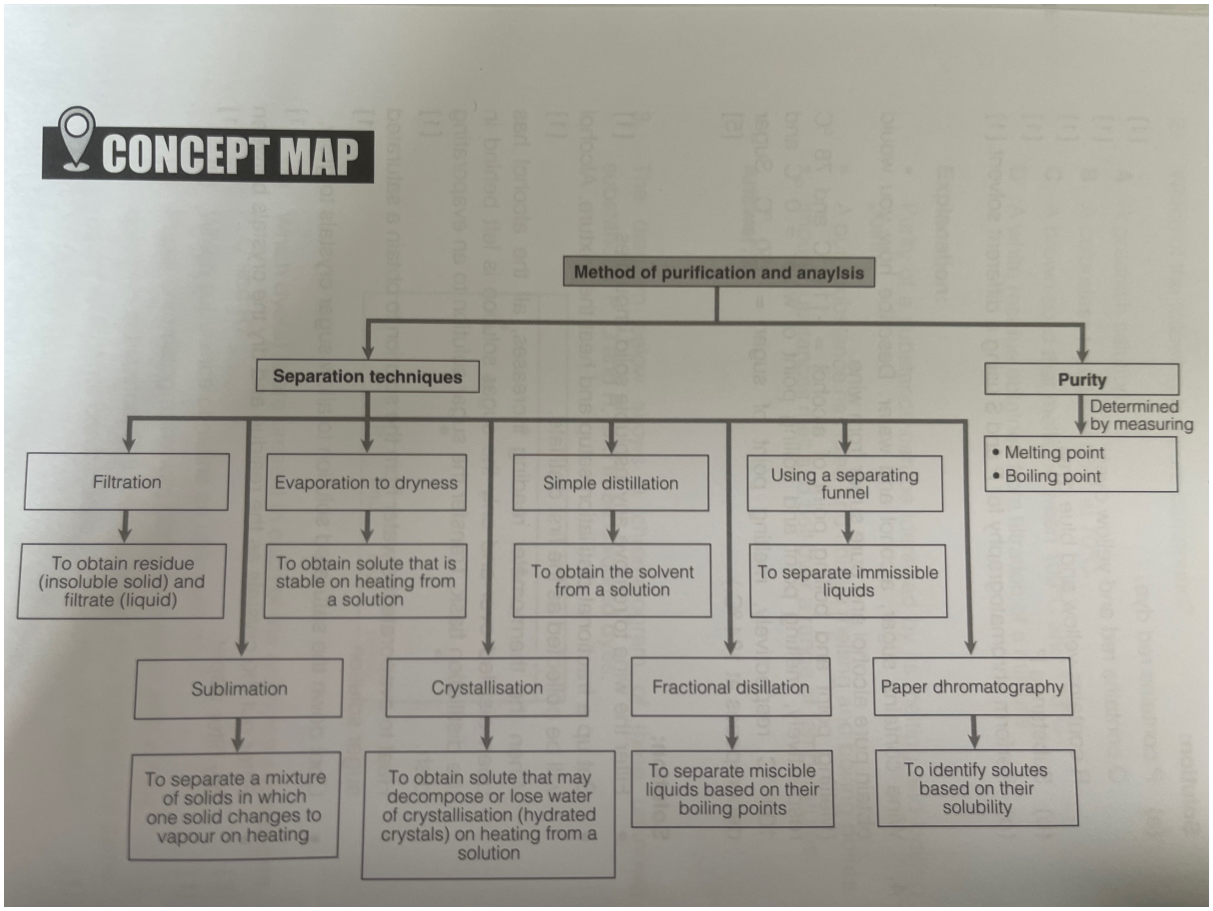
(c) Perform chromatography for P and S using a different solvent. [1]

4. Wine contains sugar, alcohol and water. Describe how you would obtain pure alcohol and pure sugar from wine.

(Melting point and boiling point of alcohol = -114 °C and 78 °C respectively, melting point and boiling point of water = 0 °C and 100 °C respectively, melting point of sugar = 150 °C. Sugar decomposes at 184 °C.) [5]

Solution:

- Filter the wine to remove any insoluble solid impurities. [1]
- Set up a fractional distillation setup and heat the mixture. Alcohol will be collected as the first distillate. [1]
- When the thermometer reading increases, all the alcohol has been distilled over and only the sugar solution is left behind in the distillation flask. Transfer the sugar solution to an evaporating dish. [1]
- Heat to evaporate the water from the solution to obtain a saturated sugar solution. [1]
- Cool down the saturated solution to allow sugar crystals to grow. [1]
- Filter out the crystals as the residue and dry the crystals between dry filter paper. [1]


CONCEPT MAP
Method of purification and anaylsis
Separation techniques
Purity
Determined by measuring
• Melting point
• Boiling point
Filtration
To obtain residue (insoluble solid) and filtrate (liquid)
Evaporation to dryness
To obtain solute that is stable on heating from a solution
Simple distillation
To obtain the solvent from a solution
Using a separating funnel
To separate immissible liquids
Sublimation
To separate a mixture of solids in which one solid changes to vapour on heating
Crystallisation
To obtain solute that may decompose or lose water of crystallisation (hydrated crystals) on heating from a solution
Fractional disillation
To separate miscible liquids based on their boiling points
Paper dhromatography
To identify solutes based on their solubility