5072 CHEMISTRY (NEW PAPERS WITH SPA)
TOPIC 1: EXPERIMENTAL CHEMISTRY

5067 CHEMISTRY (NEW PAPERS WITH PRACTICAL EXAM)
TOPIC 1: EXPERIMENTAL CHEMISTRY

SUB-TOPIC 1.2
METHODS OF PURIFICATION AND ANALYSIS

LEARNING OUTCOMES
a) Describe methods of separations and purification for the components of the following types of mixtures: i. Solid-Solid ii. Solid-Liquid iii. Liquid-Liquid (Miscible and Immiscible) Techniques to be covered for separations and purification include: i. Use of a suitable solvent, filtration and crystallisation or evaporation ii. Sublimation iii. Distillation and Fractioanl Distillation iv. Use of a separating funnel v. Paper Chromatography b) Describe paper chromatography and interpret chromatograms including comparison with 'known' samples and the use of R_f values c) Explain the need to use locating agents in the chromatography of colourless compounds d) Deduce the given melting and boiling points the identities of substances and their purity e) Explain that the measurement of purity in substances used in everyday life, e.g. foodstuffs and drugs, is important

A Introduction

- A pure substance is made up of only one substance and is not mixed with other substances
  - It contains only one type of atom or molecule
  - E.g. Computer chips are made from very pure crystals of silicon

- However, in nature, many substances are not pure.
  - E.g. The air that we breathe is not really pure. Air is a mixture of Nitrogen (78%), Oxygen (21%), Carbon Dioxide (2.03%) and other rare gases (0.97%). You will learn more about this in Topic 10: Air.

- A mixture is made up of two or more substances
  - It contains two or more atoms or molecules

- Mixtures can be easily separated into pure substances by a process called purification. It is done using physical methods without chemical reactions.

B Importance of Purity

Industry	Reason
High Precision Engineering Industry	- Computer chips are made from very pure silicon chips. - Even small amounts of impure substances can greatly reduce the effectiveness of a component in an electronic device.
Pharmaceutical Industry	- Impurities in medicines can produce undesirable side effects. - Chemists need to work with pure substances because they retain their original properties
Food and Beverage Industry	- Preservatives and dyes are added into foodstuff and beverages to make them last longer, taste better or look more attractive - Guidelines are followed to ensure that they contain the right amount of chemicals that are safe for consumption.

C Assessing Purity of Substances

- Modern laboratories use many sophisticated instruments to determine the purity of a substance.

- In a school laboratory, three simple methods are used:
  - Melting Point Determination
  - Boiling Point Determination
  - Chromatography

- The melting and boiling points of pure substances are unique. (No two substances share the same pair of melting & boiling point values)

- Hence, a pure substance can be identified from its melting and boiling points.

	Solids		**Liquids**	
Pure	Sharp and constant melting point Melts constantly at one temperature or very narrow range of temperature (< 0.5^0C)		Sharp and constant boiling point Boils constantly at one temperature or very narrow range of temperature (< 0.5^0C)	
How impurities affect it	**Lowers Melting Point** (↑impurities → ↓melting point)	Pure ice melts at 0^0C, while Frozen seawater melts around -2.5^0C	**Increases Melting Point** (↑impurities → ↑ boiling point)	Pure water boils at 100^0C, while seawater boils at around 102^0C
	Causes Melting to take over wider range of Temperatures	Coconut oil starts melting at 14^0C, and completes melting at 22^0C	**Causes Melting to take over wider range of Temperatures**	Petrol fuel starts boiling at 35^0C and completes boiling at 75^0C
Determining the Purity	**Melting Point Determination** (Refer to previous notes)		**Boiling Point Determination** (Refer to previous notes)	

II Chromatography

- Chromatography is a technqiue which can be used:
  - To separate the components in a sample and, hence, to identify the number of substances in it.
  - To identify the substances present in a sample.
  - To determine if the sample is pure.
  - To separate and identify mixtures of organic compounds such as coloured dyes, inks and foods.
    - Check that approved artificial dyes are present in foodstuff.
    - Check that the right amount of pesticides are sprayed on vegetables.
  - To test for presence of illegal drugs in athletes

- Pure substances form one “spot” on a chromatogram.

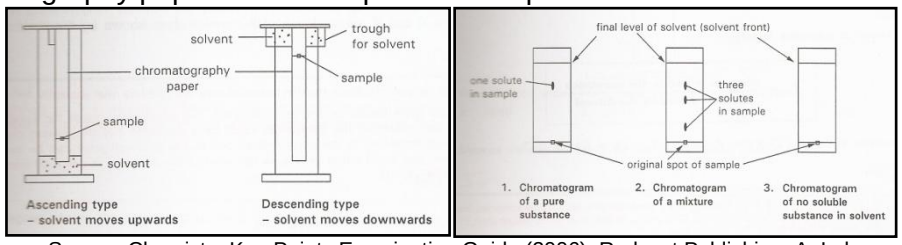
- Impure Substances/ Mixtures form two or more “spots” on a chromatogram.

- The simplest type of chromatography is called paper chromatography.

- The more advanced types of chromatography are:

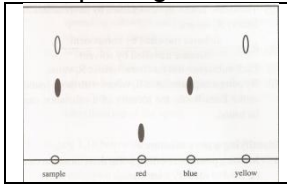
- TLC (Thin Layer Chromatography)
- HPLC (High Performance Liquid Chromatography)
- GC (Gas Chromatography)

- A chromatogram is the chromatography paper with the separated components.



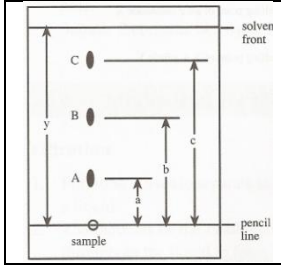
Source: Chemistry Key-Points Examination Guide (2006), Redspot Publishing, A. Loh

- Interpreting the Chromatogram:



- Identicial dyes travel the same distance up the paper.
- The components of black ink can be identified by comparing them with the known dyes.
- Black ink is made up of two different dyes.
- They are: Blue and Yellow ink dyes.
- There is no red dye present in black ink.

- Analysing the Chromatogram:



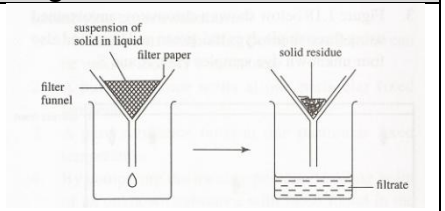
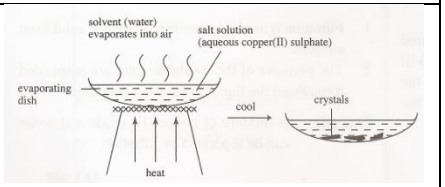
- The R_f value of a substance is the ratio of the distance travelled by the substance to the distance travelled by the solvent.

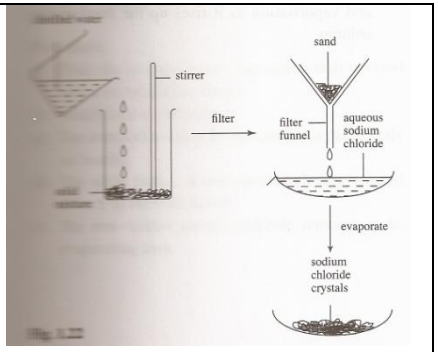
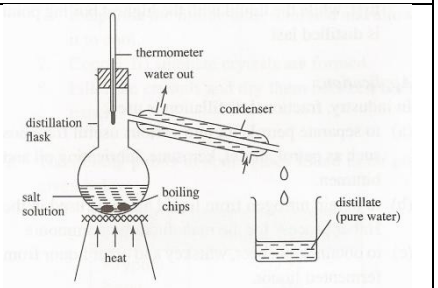
  $R_f = \frac{\text{Distance travelled by the substance}}{\text{Distance travelled by the solvent}}$, where $R_f > 1$
- The R_f value is a constant, given the same conditions (i.e. the same solvent and temperature conditions)
- In the diagram on the left, the R_f values of A, B, and C are $\frac{a}{y}$, $\frac{b}{y}$ and $\frac{c}{y}$ respectively.

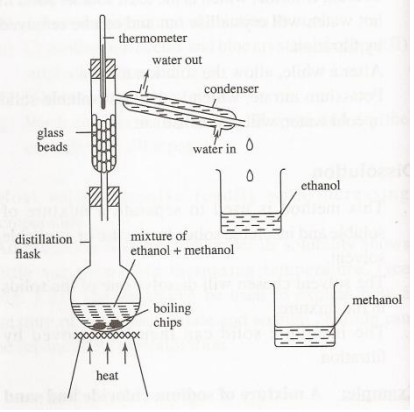
- Points to note about Chromatography
  - The starting line should be marked with pencil and not ink as the dyes in the ink may be soluble in the solvent, affecting the results of the chromatogram.

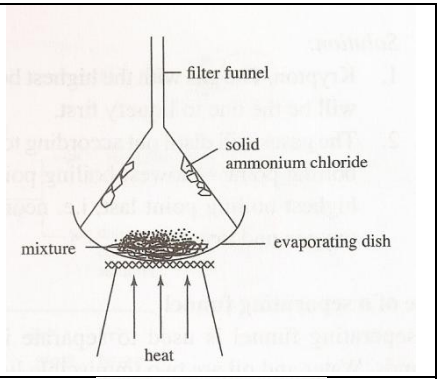
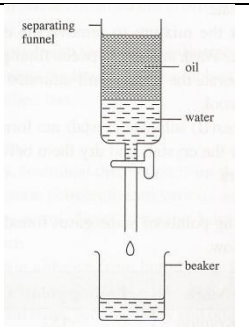
- The initial solvent level must be below the starting line. Otherwise, the sample would dissolve directly into the solvent, resulting in a poor chromatogram.
- The solvent should be allowed to run as far up as the paper as possible to ensure better separation.
- When the components of a sample are colourless, a locating agent is used to make a chromatogram visible.

D Methods of Purification

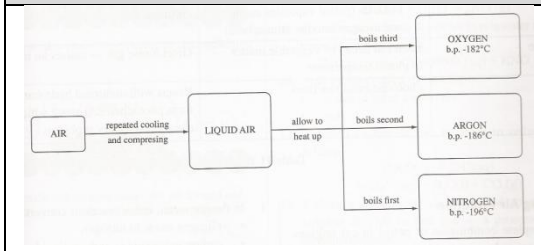
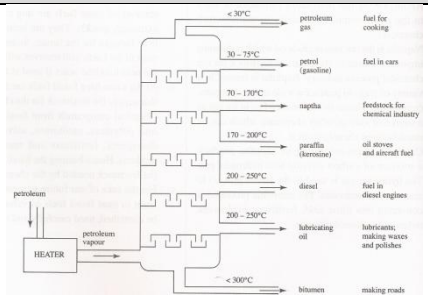
Method	Procedure	Remarks	Diagram
Filtration E.g. Mixture of Copper (II) Oxide and Water	- Fold a piece of filter paper and fill it into a filter funnel. - Wet the filter paper with water. - Pour the mixture into he filter funnel. - The insoluble Copper (II) Oxide remains in the filter funnel as residue. - The liquid water passes through the filter paper and is collected as filtrate.	- Filtration is used to separate an insoluble solid from a liquid. - The particles of the insoluble solid are suspended throughout the liquid to form a suspension.	
Crystallisation E.g. Crystals of Copper (II) Sulphate	- The aqueous Copper (II) Sulphate solution is poured into an evaporating dish. - The solution is heated to evaporate off some water. - Continue heating until the solution becomes saturated. - Allow the hot saturated solution to cool. - Crystallisation occurs and the blue crystals of Copper (II) Sulphate are formed. - Filter to remove the crystals. - Wash crystals with a little cold water and dry crystals with filter paper.	- Crystallisation is used to separate a soluble solid from its solution. - This method is based on the different solubilities of solids in water. - The solubilities of most solids increase rapidly as the temperature rises. - Hence when a saturated solution is cooled, the solubility of the solid will decrease and the excess solid will be crystallised out.	

Dissolution E.g. A mixture of Sodium Chloride and Sand	- Add distilled water to the mixture and stir. - Sodium Chloride will dissolve in the water whereas Sand will not dissolve. - Filter the mixture. - The insoluble Sand remains in the filter funnel as residue. - The soluble Sodium Chloride passes through the filter paper and is collected as filtrate. - The filtrate is then evaporated to dryness to obtain the Sodium Chloride.	- This method is used to separate a mixture of soluble and insoluble solids by the use of a suitable solvent. - The solvent chosen will dissolve one of the solids in the mixture. - The insoluble solid can then be removed by filtration.	
Distillation E.g. Pure Water from a salt solution	- Pour the salt solution into a round bottomed flask. - The distillation flask should not be more than half full. - Add a few pieces of boiling chips to smooth the boiling (i.e. to smooth the vaporisation of the liquid). - Ensure the bulb of the thermometer is opposite to the side arm exiting from the flask. - Heat the flask on a wire gauze or sand tray so that that is not concentrated on one spot. - Heat the solution until it boils. - The water vaporises. - The vapour rises up the flask and enters the condenser. - In the condenser, the water vapour condenses into liquid water. - Pure water is collected as the distillate in a collecting vessel.	- Simple distillation is carried out to separate a volatile liquid from a non-volatile solute. - On heating, the volatile liquid boils and turns into vapour. - In the condenser, the vapour condenses back into a pure liquid. - Hence the volatile liquid will distil over as the distillate. - The non-volatile liquid has a much higher boiling point as compared to the volatile liquid and will not distil over during the distillation. - The non-volatile solute remains as residue in the distillation flask. - In a simple distillation, the boiling and condensation process occurs only once.	

	- The collecting vessel must not be sealed to the condenser to avoid an explosion. - The salt remains in the round bottom flask.		
Fractional Distillation E.g. A mixture of Methanol (boiling point 65^0C) and Ethanol (boiling point 78^0C)	- The liquid mixture is poured into the distillation flask as shown. - Add a few pieces of boiling chips. - The liquid mixture is heated and brought to boil. - Both Methanol and Ethanol change to vapour. - The more volatile Methanol vaporises more easily producing more Methanol vapour in the vapour phase. - The vapour rises up the fractionating column. - The glass beads provide a larger surface area so that vapour can undergo repeated condensation and vaporisation as it rises up the fractionating column. - The first vapour to reach the top of the fractionating column is the more volatile one (i.e. the liquid with the lowest boiling point). - Methanol will distil over when the thermometer reads a constant 65^0C - When the entire Methanol has distilled over, the temperature will rise again. - The receiver is changed to collect Ethanol which will distil over at 78^0C.	- Fractional distillation is used to separate two or more miscible liquids with different boiling points. - Miscible refers to two liquids that do mix/ dissolve in each other. - The liquids are distilled out in order of their boiling points. - The liquid with the lowest boiling point is distilled first, while the liquid with the highest boiling point is distilled last.	

Sublimation E.g. Ammonium Chloride and Sodium Chloride	- Place the mixture in an evaporating dish covered with an inverted filter funnel. - Gently heat the mixture. - The Ammonium Chloride changes into a gas directly on heating. - The white fumes of Ammonium Chloride rise and reform on the cold funnel. - The non-volatile Sodium Chloride remains in the evaporating dish.	- Sublimation is a technique used to separate a mixture of two solids, one of which sublimes.	
Separating Funnel E.g. A mixture of oil and water	- Pour the mixture into a separating funnel. - Allow the two liquids to separate into two layers – the less dense oil will form the upper layer and water, the lower layer. - Open the tap to run out the bottom water layer into a beaker. - Use another beaker to collect the Upper layer.	- A separating funnel is used to separate immiscible liquids. - Immiscible refers to two liquids that do not mix/ do not dissolve in each other.	

E Applications of Fractional Distillation

Fractional Distillation of Air	Fractional Distillation of Crude Oil	Making of Alcoholic Beverages
		- The main constituent in these beverages is ethanol, which can be prepared by the fermentation of glucose. - During fermentation, glucose is broken down by yeast (catalyst) to ethanol and carbon dioxide Glucose → Ethanol + Carbon Dioxide - The final product obtained is a dilute solution of ethanol known as fermented liquor. - Ethanol is prepared by fermented liquor by fractional distillation