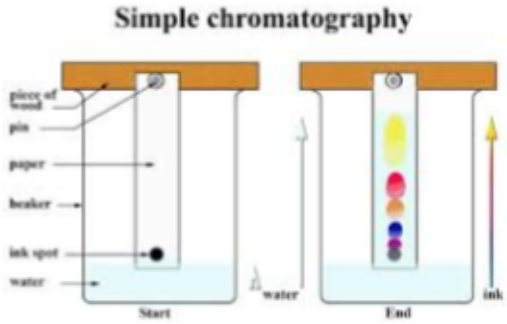
STUDY PACK : 1.2

CAMBRIDGE
International Examinations

1. Experimental Chemistry

Methods of Purification & Analysis



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Methods of Purification & Analysis

1.

i. Explain what is meant by pure substances

A pure substance consists of one substance only. It has no contaminating impurities.

ii. Explain how pure substances can be used to test purity of a sample.

A pure substance melts & boils at definite temperatures. Therefore their values for melting & boiling points are precise & predictable. Therefore we can use them to test purity of a sample & also identify an unknown substance based on their melting/ boiling points.

iii. State how the presence of an impurity in a substance effects the melting & boiling point

- Presence of impurities lowers the melting point of a substance
- And raises the boiling point of the substance.

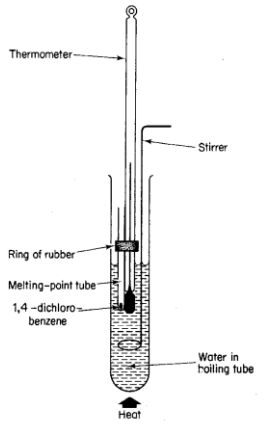
In addition, the impurity also reduces the sharpness of melting or boiling point. An impure substance melts/boils over a range of temperatures.

iv. Suggest a simple experiment to show sea water is impure.

Put some sea water in to an evaporating dish & boil away the water. A solid residue of salt will be left behind in the dish. Also sea water freezes at a temperature well below the freezing point of pure water (0^0C) & boils at a temperature above the boiling point of pure water. ($100\ ^0C$)

2. Describe & draw a detailed diagram of the apparatus which s used to measure the melting point of a solid.

The melting point of a solid can be measured using the apparatus shown.



Apparatus used for the determination of melting points

A powdered solid is put in the narrow capillary tube so that it can be heated easily. An oil bath can be used to measure melting points above 100^0C & a water bath can be used to measure melting points below 100^0C. Observe the temperature at which the solid begins to melt which is the melting point of that substance.

3. Explain the differences between mixtures & compounds

Compound:-

A Chemical compound is formed when two or more elements combine together via a chemical bond.

Mixtures:-

Mixture contains two or more substances which are not chemically bonded together. Eg: solution of NaCl contains NaCl & H_2O

4. Write the meaning of the following terms

- SOLUTE :
- SOLVENT :
- PRECIPITATE :
- SUSPENSION :
- EMULSION :

5. What are the different techniques which are used to separate mixtures & Compounds?

i. .....
ii. .....
iii. .....
iv. .....
v. .....

6. Mixtures can be formed between different substances in below given two ways. Explain them & give examples for each.

- **Homogeneous mixtures:**

Mixtures where the substances are totally mixed together and are indistinguishable.

Eg: Solutions of salts & sugars in water.
Mixtures of gases in air.
Alloy of two solid metals.

- **Heterogeneous mixtures:**

Mixtures where the substances remain separate and one substance is spread throughout the other as small particles.

Eg: suspensions of insoluble solids in water.

7. Write the different separation techniques which are used to separate the below type of mixtures.

Type of mixture	Mixture	Method of separation
Heterogeneous	Solid + solid (powdered) Suspension of solid in liquid Liquid + liquid (Immiscible)	
Homogeneous	Solution of solid in liquid Miscible liquids mixed together Solution of two (or more) solids in a liquid	

8. For each technique mentioned below give:

- The principle on which the technique is based
- An example of a mixture to which the technique can be applied
- A labeled diagram of the apparatus used
- A brief description of the procedure

i. Filtration:

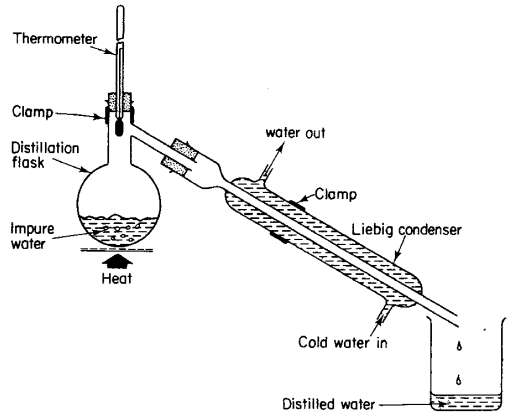
This technique is used to separate a mixture of soluble & insoluble solids, by dissolving the soluble solid in a suitable solvent & passing the mixture through a filter. Also to separate a solid from a gas filtration is used.

The principle on which this technique is based on is solubility. The filter paper consists of a porous material which prevents solid particles from passing through. The left on the filter paper is called the **residue**. The liquid that passes through the filter is called the **filtrate**.

Eg: separation of salt from sand.

ii. Distillation:

Simple distillation is designed to evaporate a volatile liquid from a solution of non-volatile / less volatile substances; the vapour is then condensed in the water condenser and collected in the receiver. Simple distillation can be used to separate a liquid from a solution (separating the solvent that dissolves substances, from a solute with greater difference in boiling points >70 ^{0}C)



Laboratory apparatus for distillation

The apparatus consists of a **round-bottomed distilling flask** connected to a **water condenser** (Liebig condenser). The thermometer bulb should be at the same level of the condenser to find the temperature of the exit vapour.

Water is sent against gravity to ensure proper cooling. The condenser has to be a water condenser if the boiling point of the liquid is below 100^0C, if above 100^0C an air condenser should be used.

If a flammable substance is heated then it should be done by a water bath/ oil bath / electrical heater.

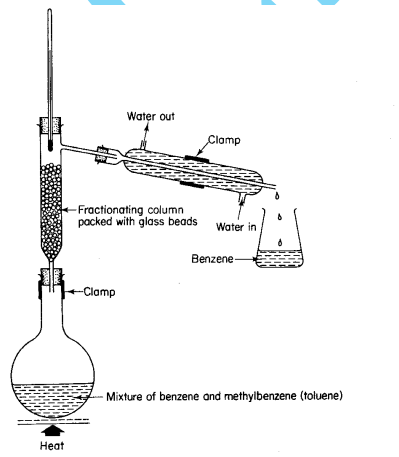
This simple distillation is used when separating a liquid, which is mixed with solids. When there is a homogeneous liquid mixture there should be significantly large difference in the boiling points of the liquids to use simple distillation. Separation of Mixtures with close boiling points (<70°C) is done by Fractional distillation.

Eg: Separation of pure water from sea water.

iii. Fractional distillation:

Fractional distillation is used to separate two or more miscible liquids based on their different boiling points. This process is similar to the distillation except that a fractionating column is fitted between the distilling flask & the condenser.

The mixture is heated, & the liquid with the lowest boiling point will boil first. This liquid vaporizes & condenses before the other liquid. The Fractionating column attached consist of glass beads packed together. The fractionating column helps to make sure that the second liquid does not get into the condenser until the entire first



Separation of a benzene/methylbenzene (toluene) mixture fractional distillation

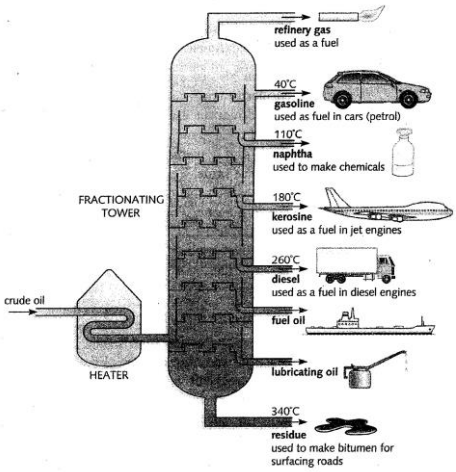
The fractionating column helps to cool the vapors rising up the column. The length of the fractionating column can be varied based on the boiling points of the liquids. If the difference in boiling points is small then a long fractionating column should be used.

The temperature of the thermometer stays constant until the relevant solution distills off. The as the second solution starts to enter the temperature of the thermometer rises. By observing the temperature difference various fractions can be separated.

Eg: Separation of ethanol (B.P 78^{0}C) from water (B.P.100^{0}C)

- Fractional distillation of **Crude oil:**

Crude oil is a complex mixture of hydrocarbons. (Molecules which contain only carbon & hydrogen) Crude oil itself is not very useful. Inorder to convert crude oil into useful products it has to be refined & this is carried out in the refinery. In the first stage of refining, crude oil is fractionally distilled based on the boiling points of each fraction.



In the refinery process, crude oil is heated to a temperature of 350-400^{0}C & pumped in at the base of the fractional distillation tower (fractionating column). As it boils the vapour passes up the tower through a series of trays & condenses as it rises further up the column at different temperatures. The smaller, lighter molecules with lower boiling point distills off first at the top & the long chain, heavier molecules with higher boiling point distills off last.

- Fractional distillation of **liquid air** :

The air is cooled to a very low temperature inorder to liquefy, the air is cleaned, dried & then compressed to a very low temperature till the air falls to a liquid. Then the liquid air is allowed to warm up. Various gases boil off at different temperatures, depending on their respective boiling points. The liquid air is fractionally distilled by warming upto -196^{0}C to obtain liquid nitrogen. Followed by Argon at -186^{0}C, then liquid oxygen at -183^{0}C.

	Simple distillation	Fractional distillation
Advantages	• Simpler setup than fractional distillation. • Faster distillation. • consumes less energy than fractional distillation	• much better separation between liquids than simple distillation • purifies readily the complex mixtures than simple distillation
Disadvantages	• requires the liquids to have large boiling point differences (>70^{o}C) • poorer separation than fractional distillation • only works well with relatively pure liquids	• more complicated setup than simple distillation • Takes longer time. • consumes more energy than simple distillation
Used for:	Separating relatively pure liquids with large boiling differences or liquids with solid impurities.	Separating complex mixtures of liquids with smaller boiling point separations.

Draw labelled diagrams of distillation & fractional distillation of a flammable substance.

Indicate the position of the thermometer, direction of water flow in the condenser, indirect heating method (oil/water bath & electric heater), Level of water/oil in the bath.

Simple Distillation:

Fractional Distillation:

iv. Crystallisation:

This technique is used to separate a solid from a solution. The solution is heated over a water bath until the point of crystallization. The point of crystallization is the temperature in which the crystals begin to appear in the mixture upon cooling. In order to determine this point a glass rod is dipped from time to time and checked for crystals upon cooling.

Once the solution is heated upto the point of crystallization it is taken off the water bath & left to cool at room temperature. Crystals begin to appear gradually upon cooling. At this point filter off the crystals & dry it in an oven or a desiccator.

v. Paper Chromatography:

Chromatography is used to separate mixtures of soluble substances. It depends on the substances having different solubilities in a solvent. This is a technique of identification of mixtures by a moving solvent on an absorbent material such as filter paper. (Chromatography paper)

A drop of concentrated solution is usually placed on the pencil line drawn at the bottom edge of a strip of filter paper. The end of the strip is then dipped in the solvent. (water or organic solvent) Solvent is selected based on their solubilities. Organic solvents dissolve many substance which are insoluble in water.
The line on the filter paper should be drawn with a pencil as ink may interfere in the experiment. Also the pencil line should be above the solvent level as shown below.

The substances separate according to their solubilities in the solvent. As the solvent moves up the paper the mixture moves with it & begin to separate. The most soluble substance will move fast up the paper. An insoluble substance will remain at the origin. Different substances in the mixture move different distance. The run is stopped just before the solvent reaches the top of the paper. (The solvent front). Spots of pure samples can be placed next to the spot of the mixture inorder to identify the mixture by comparison with the known samples.

a) Explain how R_f values are used for identification in paper chromatography?

The distance moved by a particular spot is measured and related to the position of the solvent front. The ratio of these distances is called the R_f value. (**R**atio of **f**ront) This value is used to identify the substance. Each pure substance will have its unique R_f value therefore can be identified.

$$R_f = \frac{\text{Distance moved by the substance}}{\text{Distance moved by the solvent front}}$$

b) Explain the need to use locating agents in chromatography

Locating agents are used in chromatography to identify colourless substances. The paper is treated with locating agents after the chromatography run. The locating agent reacts with the sample to produce coloured spots.

c) State the importance of chromatography in analysis

It is very useful in analysis of biologically important molecules such as sugars, amino acids & nucleotide bases.

The purity & identity of substances

9.

i. Explain how paper chromatography can be used to check the purity of a substance?

A pure sample would only produce one spot when run in several solvents. The identity of the sample can be checked by comparing its R_f values.

ii. Explain why the measurement of purity in substances is important in everyday life?

Pharmaceutical drugs must be of the highest possible degree of purity as contamination of substances may result in harmful side effects.

10. Write short notes on below seperational techniques.

i. Decantation:-

This is the first stage of separating mixtures of immiscible liquids. It is also used to separate solids in liquid. Once the solid has settled to the bottom (sediment), the liquid can be carefully poured off & this is called decantation.

ii. Separation of two immiscible liquids using a separating funnel:

The immiscible mixture is allowed to stand in a separating funnel. The liquids separate into different layers. The lower, denser layer is then tapped off at the bottom by opening the tap and the less dense top layer is poured off from the lid. This separation is based on the differences in density.

iii. Separations based on magnetic properties:

Magnetic iron ore can be separated from other materials in the crushed ore by using an electromagnet. Also in recycling, iron objects can be picked out from other metals using an electromagnet.

iv. Centrifugation:

This is another technique of separating an insoluble solid from a liquid. The mixture is spun at high speed in a centrifuge. Then centrifugal force causes solid particles to settle instead of the gravity. The centrifugal force causes the solid to be sediment at the bottom of the tube. The liquid can be then decanted off carefully.