

O-Level Chemistry

Notes

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LACAS

KIMS

Roots IVY

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Experimental Techniques

Experimental Design

Chemistry is an experimental science. We perform many experiments in chemistry. To carry out these experiments we use collections of instruments called apparatus. In this section we will discuss

- the apparatus used in chemistry lab for the measurement of volume, mass, temperature and time
- the apparatus used to collect gases and to study speed of reaction

Volumes of Liquids

The volume of a substance is the amount of space it occupies. Its units are as follows:

SI unit: cubic metre (m^3)

Other units: decimetres (dm^3)

millilitres (ml) or litres(l)

1 litre = 1 dm^3

$1 \text{ dm}^3 = 1000 \text{ cm}^3$



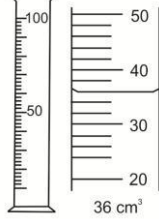

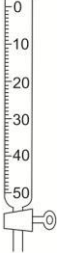

1 litre = 1000 ml

1 ml = 1 cm^3

There are many lab apparatuses used for measuring the volume of a liquid. Apparatus for measuring liquids depends on:

(i) The volume being measured

(ii) How accurate the measurement needs to be

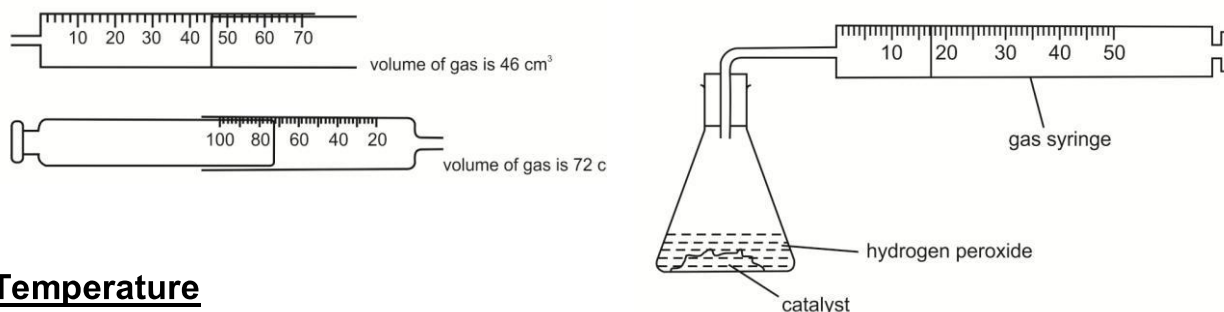
					
Beaker	Conical Flask Titration Flask	Measuring cylinder	Pipette	Burette	Measuring flask
Holds approximate volume of 100 cm^3 , 250 cm^3 , 500 cm^3 etc	Holds approximate volume of 100 cm^3 and 250 cm^3 . Used to carry out titration	Accurate to 1 cm^3 . Reading to be taken nearer to the meniscus (bottom line). If reading is 23 cm^3 , It should not be written 23.0 cm^3 as the '0' means accurate to 0.1 cm^3	Measures exact volumes such as 10.0 or 25.0 cm^3 , not odd volumes such as 11.0 cm^3 , 22.5 cm^3 . A pipette filler or safety bulb is used to fill dangerous chemicals like acids and alkalies.	Has long scale of $0 - 50 \text{ cm}^3$. Accurate to 0.1 cm^3 . It is used to take out any volume e.g. 23.6 cm^3 , 10.2 cm^3 etc. Liquid level is measured before and after opening the tap. The difference of readings gives the volume of liquid poured off.	Measures fixed volumes e.g. 100 cm^3 , 250 cm^3 , 1 dm^3 . It is used to make solutions of exact volumes.

Plastic syringe:

is used to measure small volume of liquid.

Glass syringe:

is used to measure volume of gases up to 100 cm^3 .

**Temperature**

SI Unit : Kelvin (K)

Daily life measurement: degree Celcius ($^{\circ}\text{C}$)

$$\text{K} = ^{\circ}\text{C} + 273$$

Measured with thermometer. Two types are:

- (a) Mercury – in – glass (b) Alcohol – in – glass

Temperature can also be measured using **temperature sensors or probes** with greater accuracy.

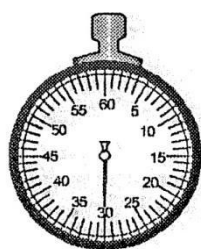
Time

SI Unit: seconds (s)

Other Units: minutes (min)/ hour (h)

Measured with

- (a) Analog stopwatch (b) Digital stopwatch



Analog stopwatch



Digital stopwatch

Analog stopwatch is accurate to nearest second. Digital stopwatch is accurate to nearest 0.01 sec.

Mass

It is the amount of matter in a substance.

SI Unit: kilogram (kg)

Other Units: grams (g)/ milligrams (mg)

$$1\text{Kg} = 1000\text{g}$$

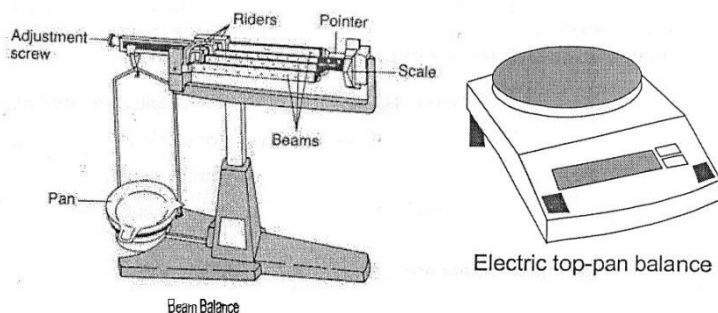
Large mass measurements: tonnes (t)

$$1\text{ tonne} = 1000\text{ kg or }10^6\text{ g}$$

Measured with:

- (a) Beam balance
- (b) Electronic “top – pan”

Electronic balance is more accurate than beam balance.



Apparatus for Collection of Gases

Method used to collect a gas depends upon two of its properties:

1. Solubility of gas in water

Solubility	Method of collection
Insoluble or slightly soluble gases (like O_2 , N_2 , H_2)	By downward displacement of water OR By displacement of air
Water soluble gases (like NH_3 , SO_2 , CO_2 , Cl_2 , HCl)	By displacement of air.

2. Density of gas

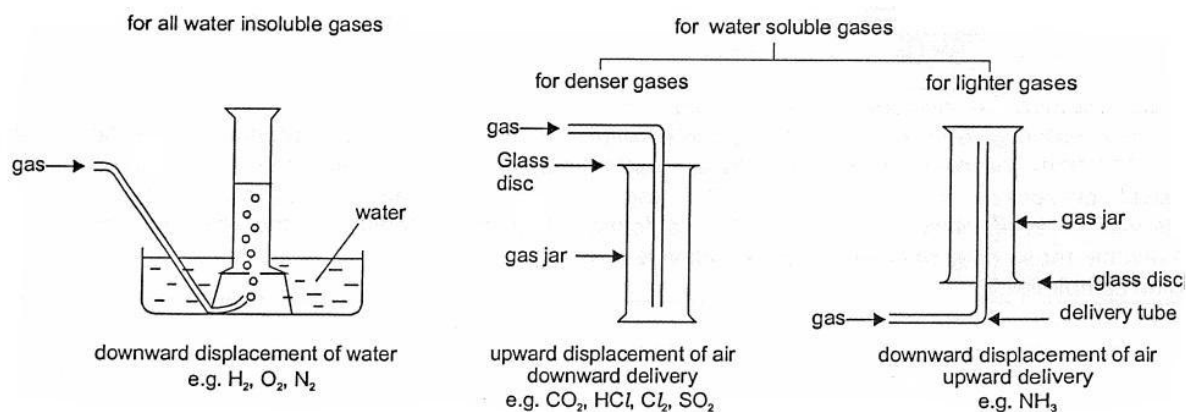
Density is directly related to relative molecular mass (M_r) are more dense.

Average M_r Air is approximately taken as 28.5.

Gases whose M_r is less than 28.5 are lighter and more than this heavier

Lighter gases (like NH_3) are collected by downward displacement of air (upward delivery).

Heavier gases (like CO_2 , SO_2 , Cl_2 , HCl) are collected by upward displacement of air (downward delivery).



During displacement with water first few bubbles of gas are rejected because it will contain air (nitrogen, oxygen etc) as an impurity.

Drying of gases

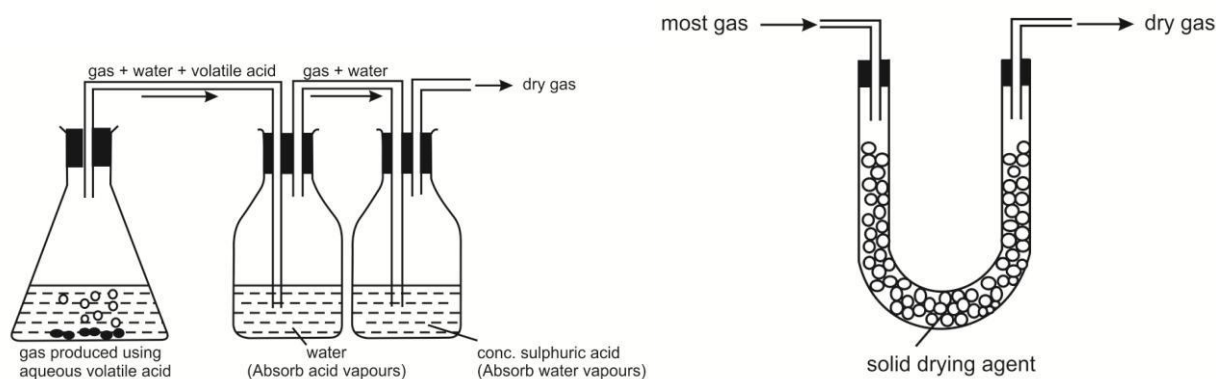
Gases prepared in laboratory may contain water vapours or the vapours of volatile acids (Acids which can evaporate e.g. HCl or HNO_3) used for their preparation. Vapours of acids are removed by passing the gas through water while water vapours are removed by passing through drying agent. Common drying agents are

conc. H_2SO_4 (for acidic or neutral gases like CO_2 , SO_2 , O_2 , H_2)

Lime, CaO or $Ca(OH)_2$ (for alkaline or neutral gases like NH_3 , O_2 , H_2)

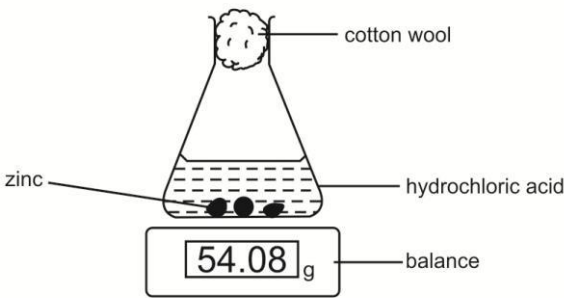
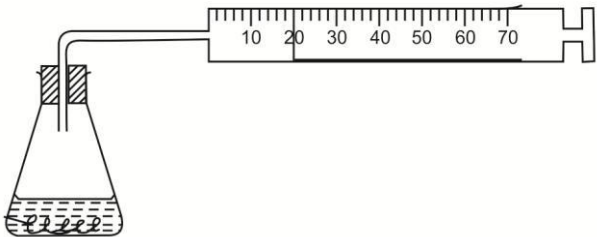
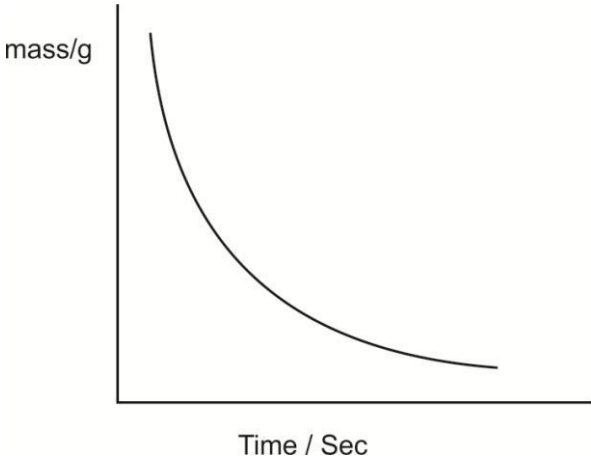
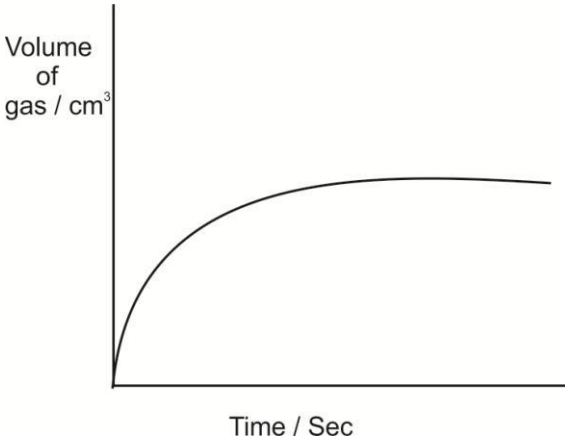
Silica gel (for any substance)

Fused $CaCl_2$ (for any substance)



Apparatus for the Measurement of speed of Reaction (or Rate of reaction)

We can determine the speed for those reactions in which some gas is formed. Speed of reaction is determined by loss in mass per unit time or volume of gas produced per unit time. Following are two ways to find reaction rates.

1. Lose in mass	2. Volume of gas collected
 <p>In this method the reaction vessel is placed on a weighing balance. Gas produced during the reaction escapes from the cotton plug. So mass of reaction vessel decreases which is noted after regular intervals of time. Hence rate of reaction is found. When the mass does not change, the reaction is said to be completed.</p>	 <p>In this method gas produced is collected in a gas syringe and its volume is noted after regular intervals of time. So rate of reaction is found. When the volume becomes constant, the reaction is said to be completed.</p>
	

Methods of Purification and Analysis

Pure substance

- Single substance not mixed with anything else
- Contain only one type of particles (atoms or molecules)

e.g: sodium chloride, distilled water, white sugar

Mixture

- Contains two or more substances. Its quantity is more on Earth.
- It contains two or more atoms or molecules.

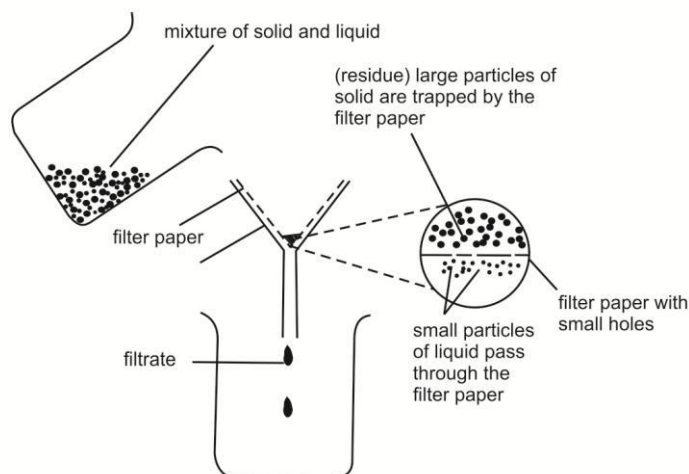
e.g: seawater (salt, water & dissolved solids), milk (fats & dissolved solids), air, petroleum

Obtaining Pure Substances

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

Filtration

- Separates insoluble solid from a liquid.
- Mixture is poured through a filter paper which has many tiny holes.
- Large solid particles cannot pass through the pores and are obtained on filter paper as residue while tiny liquid or dissolved solute particles pass through as filtrate.



Crystallization & Evaporation to Dryness

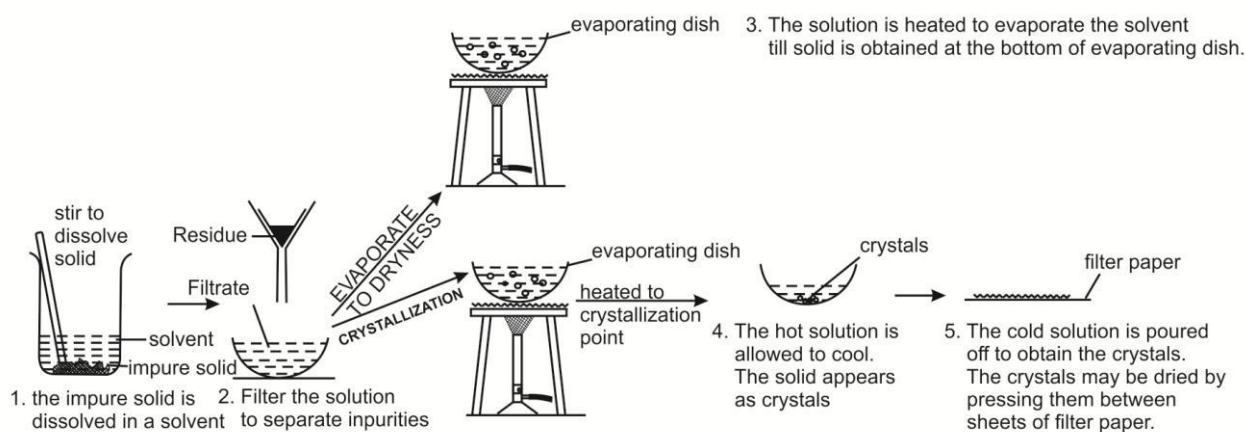
If a mixture of solids containing a soluble and insoluble solid is given then it is separated by dissolving, filtering, evaporation and crystallization.

Crystallization

- Separation of dissolved solid from a solution as well-formed crystals

Evaporation to Dryness

- Separation of dissolved solid from a solution as crystals of salt by evaporating all the liquid off.



Why crystallization occur?

Solubility of most solutes decrease as temperature decrease. When solution cools, it can't hold more solute, so the extra solute separates as pure crystals.

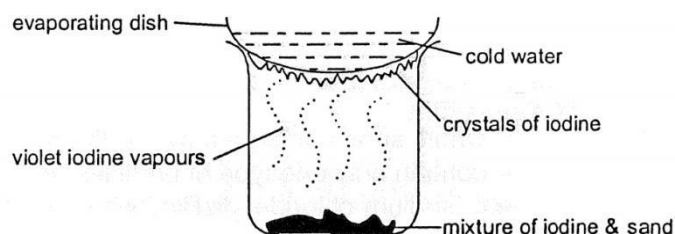
Procedure of crystallization:

- Mixture of solids is added to water. Soluble solid dissolve and the other does not.
- The insoluble substance is separated by filtration.
- The filtrate is taken in evaporating dish (china dish), which is heated to crystallization point.
- The partially evaporated solution is allowed to cool under normal conditions.
- Crystals are formed. Solvent is decanted and crystals are dried using filter paper.

In the other method the filtered solution is evaporated to dryness. But in this case care should be taken as it may damage the crystals due to removal of water of crystallization.

Sublimation

Separation of a mixture of solids, from which one solid sublimates. This is done by gently heating the solid mixture to turn one of the substances into vapour without going through liquid state. When mixture of iodine and sand is heated, iodine sublimates (turns into vapour directly) then cools and crystallize when it reaches cold water area. Non volatile sand remain in the beaker.



Examples of solids that sublime:

CO_2 (s), dry FeCl_3 (s), dry AlCl_3 (s), Naphthalene (s), I_2 (s), All ammonium salts

Simple Distillation

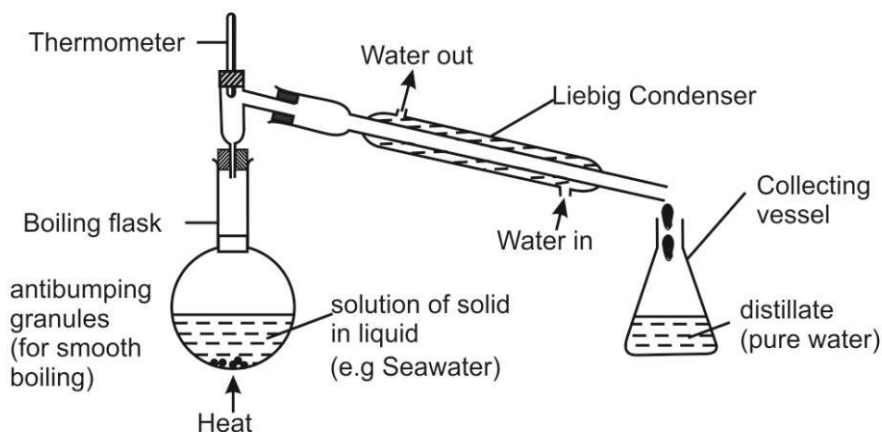
- Obtaining pure liquid from a solution by boiling the liquid and condensing the vapour.
- Solution contains dissolved solids in a liquid for example salt can be separated from sea water by distillation.
- Can be used to purify water containing insoluble impurities.

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Process of Distillation:

Solution is heated to boiling which converts the volatile liquid (i.e. liquid which can evaporate) into vapours. The vapours get cooled in condenser and form pure liquid (distillate) which is collected in receiving flask. Dissolved solutes do not evaporate due to their high boiling point and remain in the flask. Concentration of the solute in the solution, Solution increases and if distillation is allowed to proceed, non – volatile solute will be obtained in the flask as residue.

Flame is used generally to heat the flask but **electric heater** is used when the liquid is flammable.

**Important experimental details**

1. Thermometer bulb must be adjacent to the delivery tube so that it can measure the exact temperature of vapours being passed out.
2. Flow of water in condenser must be opposite to gravity so that the condenser could be fully filled. Water is entered from the bottom and it goes out from top.
3. Top of collecting vessel should be open otherwise it would be a closed system. Pressure built up can cause an explosion.

Fractional Distillation

It involves the separation of miscible liquids (liquids which mix into each other) on the basis of difference of their boiling points. Liquid with the lowest boiling point is distilled first while liquid with highest boiling point is distilled last. Fractionating column is used to separate them.

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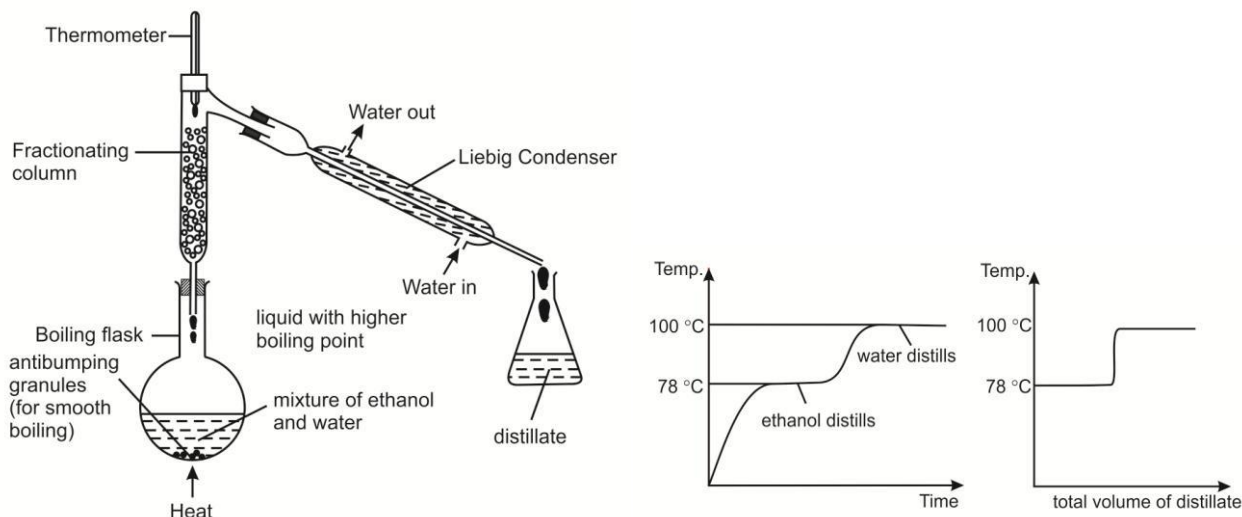
Process of Fractional Distillation:

- Pour the liquid mixture into the distillation flask.
- Add a few pieces of boiling chips to smooth the boiling (i.e. to smooth the vaporisation of the liquid).
- The liquid mixture is heated and brought to boil.
- Both water and ethanol change to vapour.
- The more volatile ethanol vaporises more easily producing more ethanol 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).
- Ethanol will distil over when the thermometer reads a constant 78°C
- When the entire ethanol has distilled over, the temperature will rise again.
- The receiver is changed to collect water which will distil over at 100°C .

Use of fractionating column: It separates the miscible liquids on the basis of difference in their boiling points.

It contains glass beads that provide a larger surface area so that vapour can undergo repeated condensation and vaporisation as it rises up the fractionating column.

Use of Condenser: It cools the vapours and converts them to liquid.



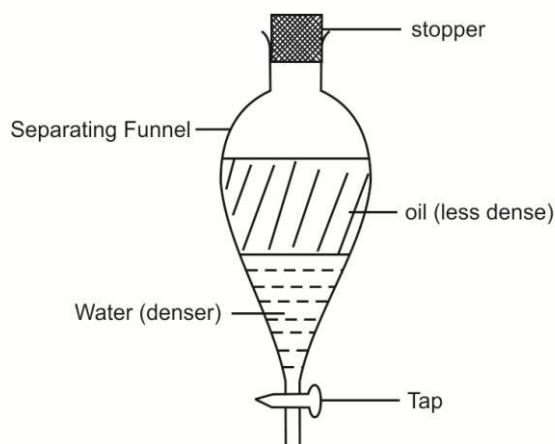
Uses of fractional distillation:

- Separates pure oxygen and pure nitrogen from liquefied air
- Separates substances in petroleum (crude oil) into fractions
- Separates alcohol from fermented liquor to produce alcoholic drinks

Use of Separating Funnel

Separating Funnel is used to separate immiscible liquids.

- 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
e.g. oil + water



Chromatography

A method of separating and identifying mixtures which may or may not be coloured.

Principle

Different solutes have different solubility into particular solvent.

The need for Chromatography

- Separates and identify mixtures of coloured substances in dyes.
- Identification and separation of amino acids.
- Separates substances in urine, drugs & blood for medicinal uses.
- To find out whether athletes have been using banned drugs.

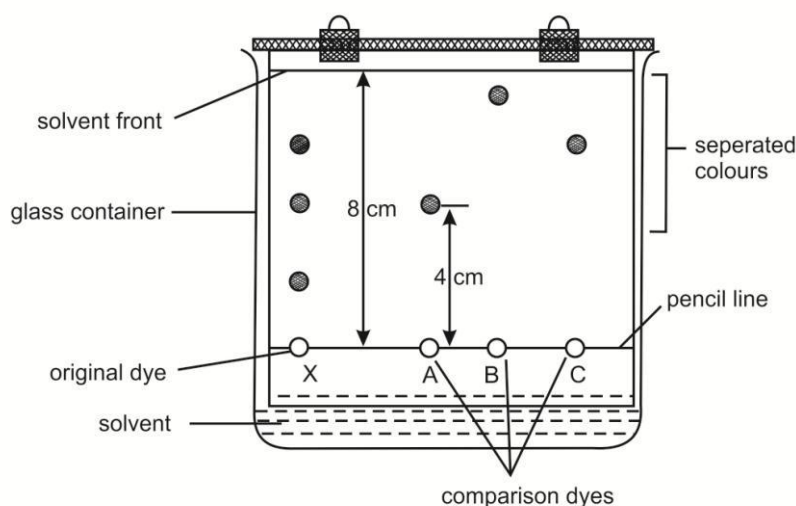
How components of a dye are separated?

- Obtain a dye (mixture) sample and put a drop of the sample on a pencil line drawn on the filter paper.
- Dip the paper into a solvent such that the spot is above solvent level.
- The solvent rises in paper.
- The dyes will dissolve in solvent. As different dyes have different solubility in the solvent so the components travel up the paper at different speeds (more soluble travels faster). Hence they are separated. A chromatogram is the chromatography paper with the separated components.

Identifying Mixtures of Coloured Substances

In the diagram on the right, drop of sample dye is placed on pencil line. The result shows that:

- The original sample dye (X) is made up of 3 colours.
- 2 comparison dyes (A & C) are present in the original dyes, as the spots have travelled equal distance.
- A comparison dye (B) isn't part of sample, as its spot do not match with any spot of sample.



Important details

1. The components must be soluble in the solvent.
2. 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.
3. While dipping the paper, the original spot must be above solvent level otherwise its components will dissolve in the solvent of container which result to a poor chromatogram.
4. The solvent should be allowed to run as far up as the paper as possible to ensure complete separation.

Separating and Identifying Mixtures of Colourless Substances

When the components of a sample are colourless, a locating agent is used to make a chromatogram visible.

Locating Agent

A substance that reacts with a substance (e.g. amino acids) on the paper to produce a coloured product.

It is used to visualize colourless components on the paper. For example ninhydrin is used as a locating agent for amino acids which produce purple spots by reacting with amino acids

R_f Values

A ratio called as retention front. A dye has constant R_f value under identical conditions.

$$R_f \text{ value} = \frac{\text{displacement travelled by component}}{\text{distance travelled by solvent}}$$

In the above diagram R_f value for dye A will be = $\frac{5}{8}$

R_f value of any component will always be less than one.

Checking the Purity of Substances

1. Pure substances have **FIXED MELTING, BOILING AND FREEZING POINTS.**

For example pure water melts at 0°C and boils at 100°C.

Impure substances have **NO FIXED MELTING AND BOILING POINTS.**

They melt and boil in a **RANGE OF TEMPERATURES** e.g. Coconut oil starts melting at 14°C, and completes melting at 22°C.

Impurity **LOWERS** the melting and **RAISES** boiling points of pure substances. e.g. Frozen sea water melts around -2.5°C and boils at 104°C.

2. Pure substance produces **only one spot on chromatographic paper.**

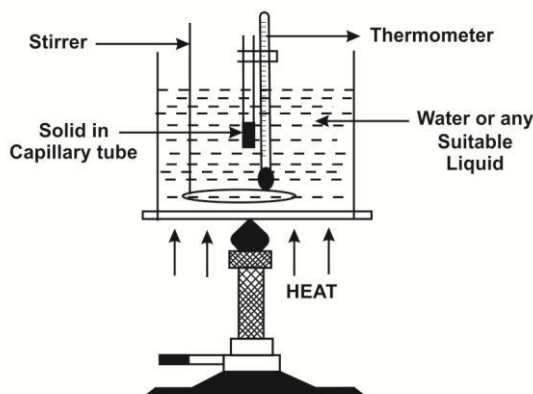
Importance of purity in food and medicine

Pharmaceutical Industry	<ul style="list-style-type: none"> ➤ 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	<ul style="list-style-type: none"> ➤ Impure food may contain toxins which can make us ill. ➤ 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.

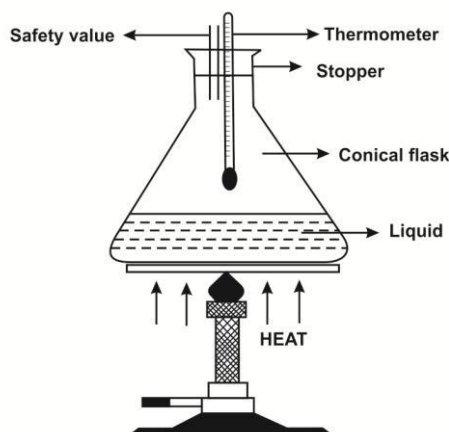
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Determining Melting Point and Boiling Point

- For solid, we find the **melting point** because pure solids have sharp (fixed) melting points. However, **impurities lower the 'melting point of pure solids**. The liquid used to heat up the solid must have a **boiling point HIGHER** than the **melting point** of the solid.



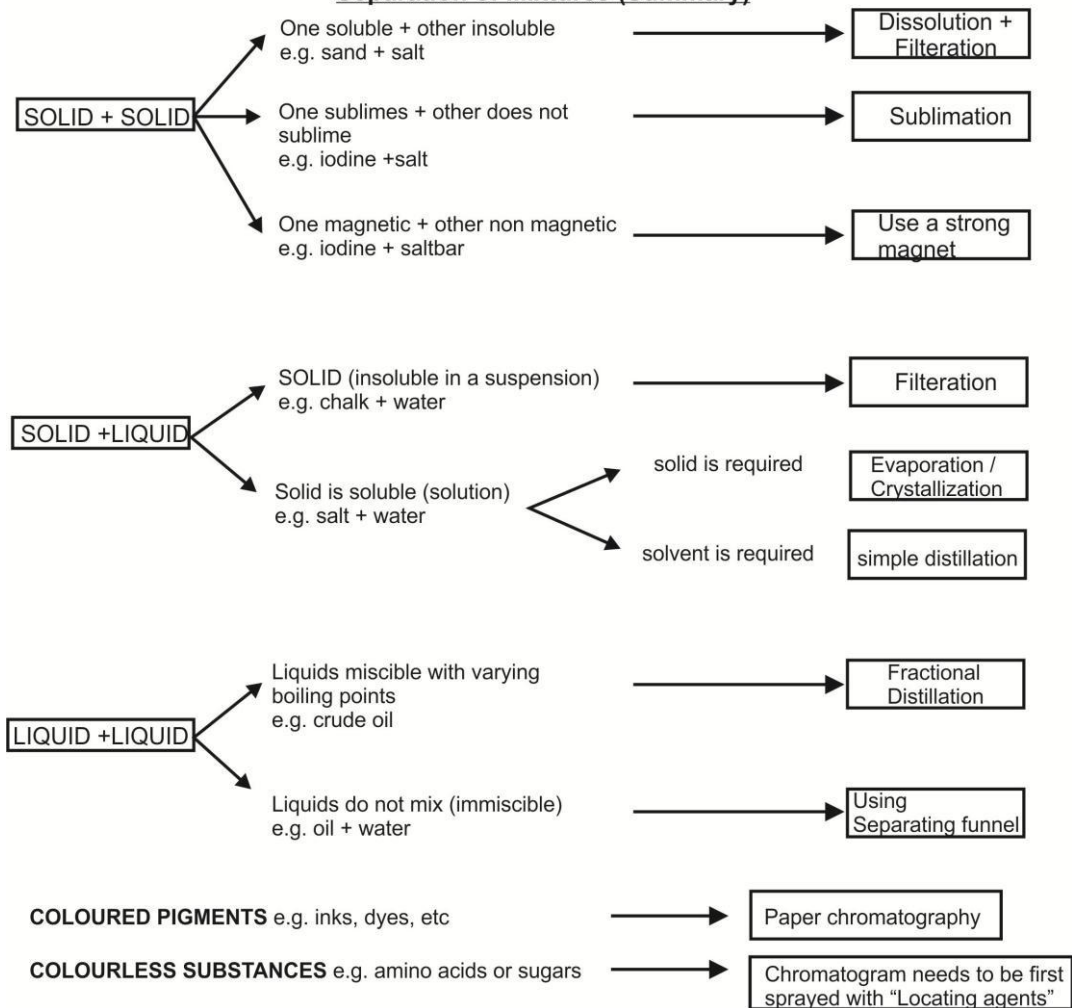
- For liquids, we find the **boiling point** because pure liquids have sharp (fixed) boiling points (boil over a very narrow range). However, **impurities raise the boiling point of pure liquids**. Tap water and sea water therefore boil at a temperature higher than 100°C.



Liquid	Melting point
Pure Water	100°C
Ethanol	78°C
Liquid Sulphur	444°C

Solid	Melting point
Pure Ice	0°C
Pure Copper	1083°C
Pure Sulphur	114°C
Pure Iron	1535°C

Separation of Mixtures (Summary)



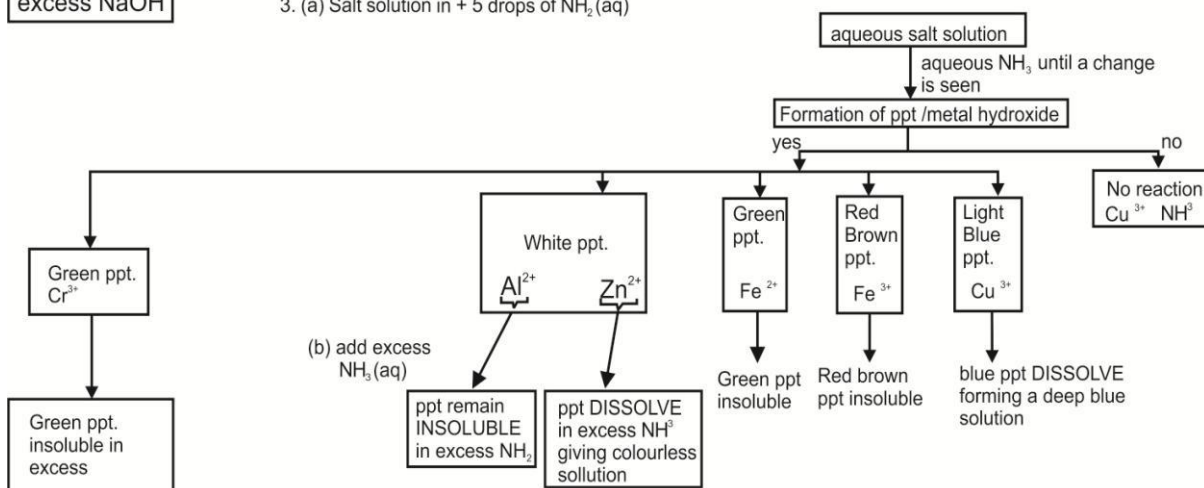
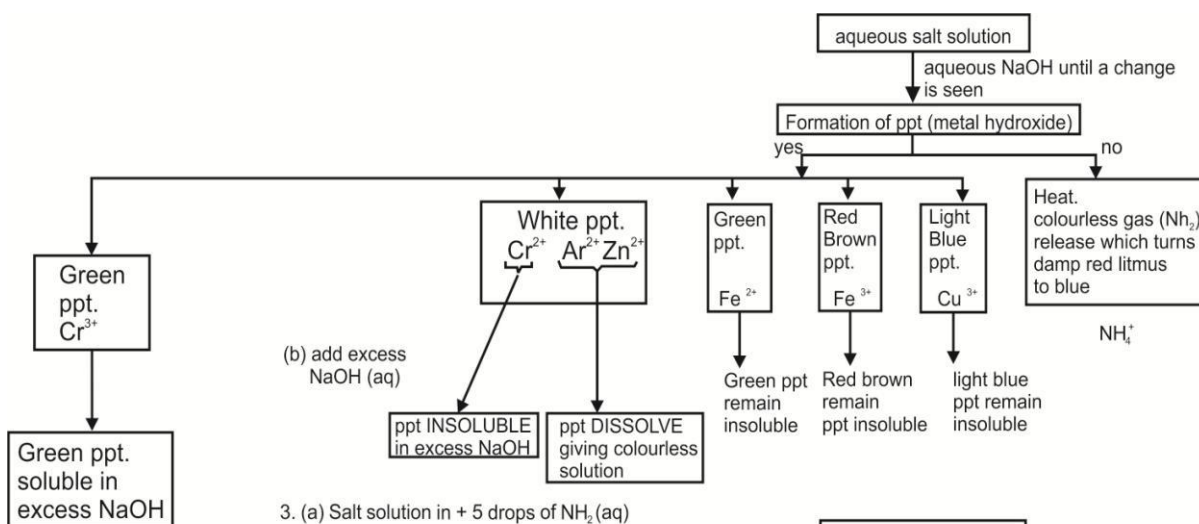
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Identification of Cations, Anions and Gases

1. Salt is dissolved in water and colour of solution is noted.

- If solution is colourless Transition metal ions are absent
- If solution is coloured Transition metal ions are present (i.e. Fe^{2+} , Fe^{3+} , Cu^{2+} , Cr^{3+})

2. (a). Salt solution +5 drops of $\text{NaOH}(\text{aq})$



Composition of precipitates:

White ppt – $\text{Ca}(\text{OH})_2$, $\text{Al}(\text{OH})_3$, $\text{Zn}(\text{OH})_2$

Blue ppt – $\text{Cu}(\text{OH})_2$,

Green ppt – $\text{Fe}(\text{OH})_2$, $\text{Cr}(\text{OH})_3$

Red brown ppt – $\text{Fe}(\text{OH})_3$

Both $\text{NaOH}(\text{aq})$ and $\text{NH}_3(\text{aq})$ contain OH^- ions. They react with cations to form precipitates as shown in the following reactions.

$\text{Ca}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Ca}(\text{OH})_{2(\text{s})}$	White ppt
$\text{Zn}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Zn}(\text{OH})_{2(\text{s})}$	White ppt
$\text{Al}^{3+}_{(\text{aq})} + 3\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Al}(\text{OH})_{3(\text{s})}$	White ppt
$\text{Cr}^{3+} + 3\text{OH}^{-} \rightarrow \text{Cr}(\text{OH})_3$	Green PPT
$\text{Cu}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Cu}(\text{OH})_{2(\text{s})}$	Blue ppt
$\text{Fe}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Fe}(\text{OH})_{2(\text{s})}$	Dirty green ppt
$\text{Fe}^{3+}_{(\text{aq})} + 3\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Fe}(\text{OH})_{3(\text{s})}$	Red Borwn ppt

Identification of anions

	Test	Observation
NO_3^{1-}	Add DIL SODIUM HYDROXIDE and ALUMINUM powder + Heat	Colourless gas (i.e. NH_3) releases which turns moist RED litmus paper to BLUE
CO_3^{2-}	Add DIL HYDROCHLORIC ACID	Carbon dioxide (CO_2) is formed with effervescence
Cl^{-1}	Add dil nitric acid (destroys any carbonate) and then add SILVER NITRATE solution	WHITE PPT of silver chloride formed which turns lime water milky
SO_4^{2-}	Add dil nitric acid (destroys any carbonate) and then add BARIUM NITRATE solution	WHITE PPT of barium sulphate formed $\text{Ba}^{2+} + \text{SO}_4^{2-} \rightarrow \text{BaSO}_4$
I^{-}	Add dil nitric acid (destroys any carbonate) and then add LEAD NITRATE solution	YELLOW PPT of lead iodide formed $\text{Pb}^{2+} + 2\text{I}^{-} \rightarrow \text{PbI}_2$
Br^{-}	Add dil. Nitric acid (destroy any carbonate) and then add silver nitrate solution	CREAM PPT of silver bromide formed $\text{Ag}^{+} + \text{Br}^{-} \rightarrow \text{AgBr}$

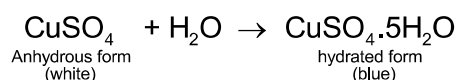
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Identification of gases

Gas	Test	Observation
Hydrogen (H ₂)	Took a BURNING SPLINT in the gas jar containing hydrogen.	Hydrogen burns with a POP sound
Oxygen (O ₂)	Took a GLOWING SPLINT in the gas jar containing oxygen.	Oxygen relights the glowing splint
Ammonia (NH ₃)	Took a moist RED litmus in the gas jar containing ammonia.	Ammonia turns moist RED litmus to BLUE.
Chlorine (Cl ₂)	Took a moist BLUE litmus in the gas jar containing chlorine.	Chlorine BLEACHES the litmus paper.
Sulphur dioxide (SO ₂)	Passed the gas through acidified solution of K ₂ Cr ₂ O ₇ .	Turns ORANGE K ₂ Cr ₂ O ₇ solution to GREEN.
Hydrogen chloride (HCl)	Took a moist BLUE litmus in the gas jar containing hydrogen chloride.	Turns moist BLUE litmus to RED.

Chemical test of Water

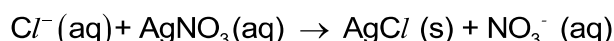
Put a drop of water on anhydrous copper sulphate (CuSO₄). White copper sulphate will change to blue.

**EXTRA INFORMATION****Why to add dil HNO₃ to identify Chloride/ Bromide/ Iodide ions?**

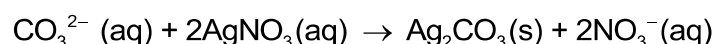
The purpose is to remove interfering ions particularly carbonates.

If you have an unknown and you test for chlorides (or halides in general) this is done by adding a solution of AgNO₃.

If a white precipitate occurs you conclude that chloride ion is present.



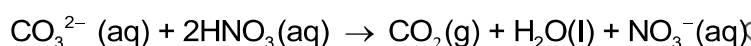
But what if the unknown contains a carbonate? The following occurs:



This will also form a white precipitate.

This is then a false positive for chlorides

To avoid this, the sample solution is first acidified with HNO₃



The CO₃²⁻ ion has been removed as an interfering anion, and any precipitate can be taken as confirming the presence of Cl⁻ ion in solution.