



1 Experimental chemistry

Content

- 1.1 Experimental design
- 1.2 Methods of purification and analysis
- 1.3 Identification of ions and gases

Learning Outcomes

Candidate should be able to:

- (a) name appropriate apparatus for the measurement of time, temperature, mass and volume, including burettes, pipettes, measuring cylinders and gas syringes
- (b) suggest suitable apparatus, given relevant information, for a variety of simple experiments, including collection of gases and measurement of rates of reaction
- (c) describe methods of purification by the use of a suitable solvent, filtration and crystallisation, distillation (including the description but not use of fractional distillation - refer to the fractional distillation of crude oil and fermented liquor).
- (d) suggest suitable methods of purification, given information about the substances involved
- (e) describe paper chromatography and interpret chromatograms
- (f) deduce from the given melting point and boiling point the identities of substances and their purity
- (g) describe the use of aqueous sodium hydroxide and aqueous ammonia to identify the following aqueous cations: aluminium, ammonium, calcium, copper(II), iron(II), iron(III), lead(II) and zinc (formulae of complex ions are **not** required)
- (h) describe tests to identify the following anions: carbonate (by the addition of dilute acid and subsequent use of limewater); chloride (by reaction of an aqueous solution with nitric acid and aqueous silver nitrate); iodide (by reaction of an aqueous solution with nitric acid and aqueous lead(II) nitrate); nitrate (by reduction with aluminium and aqueous sodium hydroxide to ammonia and subsequent use of litmus paper) and sulphate (by reaction of an aqueous solution with nitric acid and aqueous barium nitrate)
- (i) describe tests to identify the following gases: ammonia (using damp red litmus paper); carbon dioxide (using limewater); chlorine (using damp litmus paper); hydrogen (using a burning splint); oxygen (using a glowing splint) and sulphur dioxide (using acidified potassium dichromate(VI))

* - Asterisks placed alongside topics indicate **critical areas** which are not covered in the syllabus, but are included to make the learning of the topics complete.

1.1 Experimental design

SI Units

Physical quantities are used to obtain accurate measurements in experiments.

A **base quantity** is chosen and arbitrarily defined, rather than being derived from a combination of other *physical quantities*.

✎ There are seven **base quantities**:

- Length
- Mass
- Time
- Electric current
- Thermodynamic temperature
- Amount of substance
- Luminous intensity.

A **unit** is a measure of *quantity*. Measurements are given a unique unit and symbol according to the *International System of Units* or *SI*.

✎ The SI is founded on seven *SI base units* for the seven *base quantities*:

- The *base units* for these *SI base quantities* are shown in the table below:

Base Quantity	SI Base Unit	
	Name	Symbol
Length	meter	m
Mass	Kilogram	kg
Time	second	s
Electric Current	ampere	A
Thermodynamic Temperature	kelvin	K
Amount of Substance	mole	mol
Luminous Intensity	candela	cd

✎ Other quantities, called *derived quantities*, are defined in terms of the seven *base quantities* via a system of quantity equations. The *SI derived units* for these *derived quantities* are obtained from these equations and the seven SI base units.

Examples

- The following are examples of derived quantities and derived units.

Derived Quantity	Derived Unit	
	Name	Symbol
area	square metre	m ²
volume	cubic metre	m ³
speed, velocity	Metre per second	m/s
acceleration	metre per second squared	m/s ²
mass density	kilogram per cubic metre	kg/m ³
specific volume	cubic metre per kilogram	m ³ /kg
amount-of-substance concentration	mole per cubic metre	mol/m ³
mass fraction	kilogram per kilogram, which may be represented by the number 1	kg/kg = 1



- 📄 For ease of understanding and convenience, some SI derived units have been given special names and symbols. These derived units can be further included in other derived units.

Examples

- ① The following are examples of commonly used derived quantities and derived units.

Derived quantity	SI derived unit			
	Name	Symbol	Expressed in terms of other SI units	Expressed in terms of SI base units
pressure, stress	pascal	Pa	N/m ²	m ⁻¹ ·kg·s ⁻²
energy, work, quantity of heat	joule	J	N·m	m ² ·kg·s ⁻²
Celsius temperature	degree Celsius	°C	-	K



- ☺ The derived unit with the special name degree Celsius and special symbol °C deserves comment. Because of the way temperature scales used to be defined, it remains common practice to express a thermodynamic temperature, symbol T , in terms of its difference from the reference temperature $T_0 = 273.15$ K, the ice point. This temperature difference is called a Celsius temperature, symbol t , and is defined by the quantity equation

$$t = T - T_0$$

The unit of Celsius temperature is the degree Celsius, symbol °C. The numerical value of a Celsius temperature t expressed in degrees Celsius is given by

$$t(^{\circ}\text{C}) = T(\text{K}) - 273.15$$

It follows from the definition of t that the degree Celsius is *equal* in magnitude to the kelvin, which in turn implies that the numerical value of a given temperature difference or temperature interval whose value is expressed in the unit degree Celsius (°C) is equal to the numerical value of the same *difference* or *interval* when its value is expressed in the unit kelvin (K). Thus, *temperature differences* or *temperature intervals* may be expressed in either the degree Celsius or the kelvin using the *same* numerical value. For example, the Celsius temperature difference Δt and the thermodynamic temperature difference ΔT between the melting point of gallium and the triple point of water may be written as $\Delta t = 29.7546$ °C = $\Delta T = 29.7546$ K

- ❷ The following are examples of the above derived units used in other derived units.

Derived Quantity	Derived Unit	
	Name	Symbol
heat capacity, entropy	joule per kelvin	J/K
specific heat capacity, specific entropy	joule per kilogram kelvin	J/(kg·K)
molar energy	joule per mole	J/mol
molar entropy, molar heat capacity	joule per mole kelvin	J/(mol·K)



- ❸ Other commonly used units can also be derived from SI units.

Derived Quantity	Derived Unit	
	Symbol	Value in SI units
minute (time)	min	1 min = 60 s
hour	h	1 h = 60 min = 3600 s
day	d	1 d = 24 h = 86 400 s
litre	L	1 L = 1 dm ³ = 10 ⁻³ m ³
metric ton or tonne	t	1 t = 10 ³ kg



SI prefixes

✎ SI prefixes are used to form *decimal multiples* and *submultiples* of SI units.

- The common *SI prefixes* for *SI units* are shown in the table below.

Factor	Name	Symbol
10^{12}	Tera	T
10^9	Giga	G
10^6	Mega	M
10^3	Kilo	k
10^2	Hector	H
10^1	Deka	da
10^0	-	-
10^{-1}	Deci	d
10^{-2}	Centi	c
10^{-3}	Milli	m
10^{-6}	Micro	μ
10^{-9}	Nano	n
10^{-12}	Pico	p

- It is important to note that the kilogram is the only SI unit with a prefix as part of its name and symbol.

Examples

- 1 10^{-6} kg = 1 mg (one milligram), but not 10^{-6} kg = 1 μ kg (one microkilogram).
- 2 Consider the highest point in Singapore, Bukit Timah Hill at the height of 166m. It can also be written in the formats below.

Height	Factor	Conversion	SI Prefix	Symbol
166,000 mm	10^{-3}	$166,000 \times 10^{-3}$ = 166 m	milli	m
16,600 cm	10^{-2}	$16,600 \times 10^{-2}$ = 166 m	centi	c
166 m	10^0	166×10^0 = 166 m	-	-
0.166 km	10^3	0.166×10^3 = 166 m	kilo	k



- Note that no matter which form you choose to express it as, they are all basically the same measurement.

Apparatus

✍ Several important quantities are commonly measured in performing experiments.

- Time

Time is measured in the unit second (s). A stop watch can be used to measure time taken for a reaction to complete.



Digital Stopwatches



Analogue Stopwatches

- Temperature

Temperature is commonly measured in degree Celsius ($^{\circ}\text{C}$). A *mercury-in-glass thermometer* or a *digital thermometer* can be used to measure changes in temperature.



digital thermometer



Mercury-in-glass thermometer

- Mass

Mass is commonly measured in grams (g). An *electronic balance* is usually used for measuring mass. Electronic balances come in a variety of designs and may take readings up to 4 decimal places accuracy.



Electronic Balance

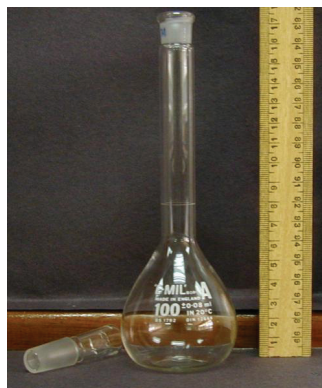
- Volume

Volume is commonly measured in cm^3 . A variety of apparatus are available for the measurement of *liquid volume* or *gas volume*

- ① Volumetric Flask

Volumetric Flasks comes in a variety of volumes. They are used to make standard solutions. All volumetric flasks

have a graduation mark on the neck of the bottle. Once the bottom of the meniscus sits on the graduation mark, the volume indicated on the volumetric flask has been reached.

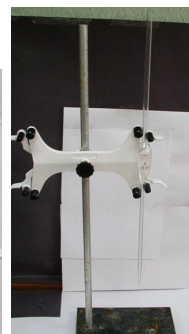


2 Pipettes

Pipettes are used to transfer an accurate volume of liquid. They come in standard sizes of 5, 10, 20, 25 and 50 cm³. A pipette filler is used to draw the liquid up the pipette. The volume required is achieved when the meniscus sits on the graduation mark.



Pipette fillers



Pipette

☺ Never use your mouth to suck liquid on a pipette. You might accidentally swallow the fluid and most fluids are hazardous to health.

3 Burette

A *burette* is a long glass tube used to measure a varying and accurate amount of liquid that is added to another liquid.



Burette with trapped air bubble



Burette with no air bubble



Burette

4 Measuring Cylinder

Measuring cylinders are used for rough measurements of liquid. They come in a variety of sizes, from as small as 1cm^3 to 1000cm^3



Glass measuring cylinder

5 Gas Syringes

Gas Syringes are usually airtight tubes with graduation markings on the side. They have a plunger to control the volume of gas inside the barrel. They also come in a variety of sizes.



Gas Syringe

Other commonly used apparatus for experiments are *conical flasks*, *round bottom flask*, *Buchner flask*, *condensers*, *separating funnel*, *Buchner funnel*, *fractionating column*, *beakers*, *clamps* and *evaporating dish*. Some of them are illustrated below.



Tripod



Beaker



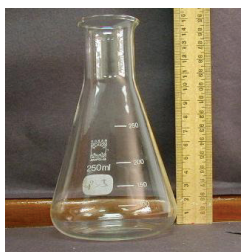
Buchner Funnel



Buchner Flask



Clamps



Conical Flask



Condenser



Delivery Tube (gas delivery)



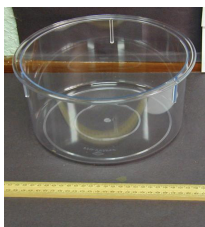
Test tube with holder



Evaporating Dish



Gas Jar



Plastic Trough



Glass Funnel



Round Bottom Flask

- ☺ All measurements are accurate to half the graduation of their smallest measurement unit. For example, for a measuring cylinder of smallest graduation of 1 cm^3 , the accuracy is to 0.5 cm^3 . Therefore, it is always wise to choose an apparatus that measure closest to the quantity required to minimize errors.
- ☺ It is important to observe good experimental practices. Glasswares and equipment should be kept clean. In particular, glasswares should be washed and dried before and after use. Residues from the previous experiments may affect your results if the apparatus are not properly cleaned.
- ☺ Distilled water or Deionised (DI) water are usually used to clean glassware. These specially prepared water are free of most ionic and organic compounds. Distilled water is obtained from distillation of tap water and Deionised water is obtained by passing tap water through a series of filters. Tap water is not used as it has a high content of chlorine ions.

Simple experimental setups

- 🔧 A variety of simple experimental setups can be done with the apparatus described. Some of the more common setups are discussed below.

Gas collection

- 🔧 A good sample of gas is obtained by proper collection and drying of the gas.

- Gas collection

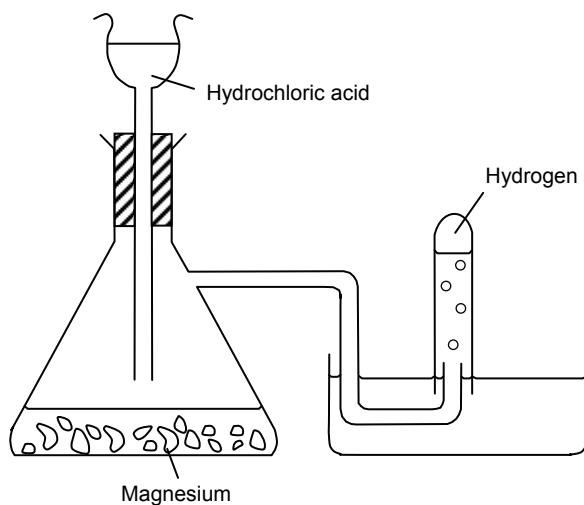
The methods of collection are shown below.

- ① Displacement of a liquid by a gas.

In this case, the gas displaced must be insoluble or almost insoluble in the liquid. A common liquid used is water

Examples

The collection of hydrogen is done as shown in the following diagram:



Hydrochloric acid is slowly added to magnesium to produce hydrogen. The resultant gas is collected over a test tube filled with water.

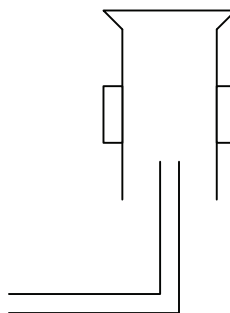


- ② Downward displacement of air

In this case, the gas collected must be less dense than air.

Examples

The collection of ammonia is done in this manner:



Ammonia is produced from the heating of sodium hydroxide with ammonium chloride. The resultant gas is collected in a glass jar.

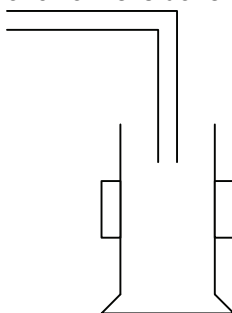


③ Upward displacement of air

In this case, the gas collected must be denser than air.

Examples

The collection of chlorine is done in this manner.



Chlorine produced from the oxidation of hydrochloric acid with potassium permanganate.

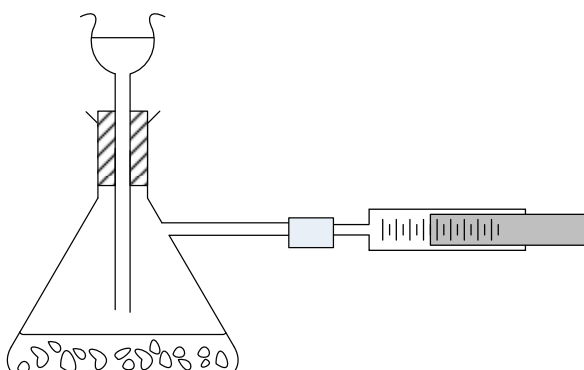


④ Gases can also be collected with a gas syringe.

This method is often used to collect a small amount of gas. It can accurately measure the amount of gas produced.

Examples

The collection of carbon dioxide is done in this manner.



Hydrochloric acid is slowly added to calcium carbonate produce carbon dioxide. The resultant gas is collected using a gas syringe.



- Drying of gases

Agents used to dry the gas must not react with the gas.

Acidic drying agents like concentrated sulphuric acid and phosphorous (V) oxide should not be use to dry alkaline gases like ammonia.

Alkaline drying agents like calcium oxide (quick lime) should not be used to dry acidic gases like chlorine, hydrogen chloride or sulphur dioxide.

Examples

The following chart shows some common gases, their appropriate drying agents and their methods of collection:

Gas	Solubility in water	Density compared with air	Drying agent used	Method of collection
Nitrogen (N ₂)	Almost insoluble	Slightly less dense	Concentrated sulphuric acid	Over water
Oxygen (O ₂)	Almost insoluble	Slightly more dense	Concentrated sulphuric acid or calcium chloride	Over water
Hydrogen (H ₂)	Almost insoluble	Very low density	Calcium chloride or phosphorous (V) oxide	Over water or downward displacement of air
Carbon monoxide (CO)	Almost insoluble	Slightly less dense	Concentrated sulphuric acid	Over water
Carbon dioxide (CO ₂)	Moderately soluble	More dense	Concentrated sulphuric acid	Over hot water or upward displacement of air
Chlorine (Cl ₂)	Soluble	More dense	Concentrated sulphuric acid	Upward displacement of air
Ammonia (NH ₃)	Very soluble	Less dense	Calcium oxide (quick lime)	Downward displacement of air
Hydrogen chloride (HCl)	Very soluble	More dense	Concentrated sulphuric acid	Upward displacement of air
Sulphur dioxide (SO ₂)	Very soluble	More dense	Concentrated sulphuric acid	Upward displacement of air



Rates of reaction

The **rate of reaction** measure the *speed* of a reaction. The higher the *rate of reaction*, the faster the speed.

- ✍ The *rate of reaction* is usually expressed in terms of the concentration (usually in moles per litre, mol l^{-1}) of a reactant consumed or product formed, in unit time. That is, in mole per litre per second ($\text{mol l}^{-1} \text{s}^{-1}$)
- ✍ The *rates of reaction* between using different reactants is used to compare the relative efficiency of a process.

- Measurements of rates of reaction

The experimental rate of reaction is usually measured either by a loss of a reactant or by the formation of a product.

① Measurement of rate of reaction by observations of loss of reactant.

- The simplest way of measuring loss of reactant is via mass loss, particularly in reactions that forms gaseous products.
- The reaction can be conducted in an open flask placed on a mass balance. As the reaction proceeds, readings are taken from the balance at 30 second intervals.
- A graph of the mass loss against time can then be plotted and the rate of the reaction estimated from the slope at any particular time instant.
- Alternatively, a fixed amount of one reactant is reacted with excess of the other to ensure that all of the limiting reactant is completely consumed. The completion of the reaction can be detected with observations of either a colour change with an appropriate indicator or the stopping of effervescence.
- The time is noted at the moment the reactants are added to each other (t_i) and the final time when no further change is observed, is also noted (t_f).
- The overall *rate of reaction* can be calculated from the formulae below:

$$\text{Rate of reaction} = \frac{\text{Amount of reactant}}{t_f - t_i}$$

where

t_i = initial time (when reactants are first added to each other)

t_f = final time (when the reaction stops)

Examples

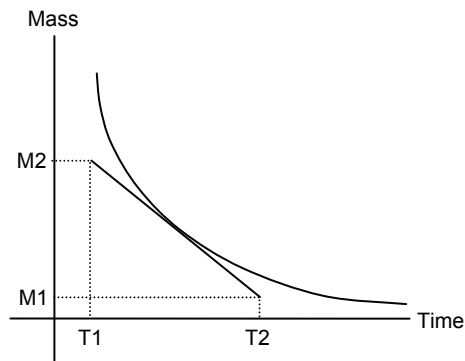
- ① Estimate the rate of reaction from the loss of reactants:

The rate of reaction of excess calcium carbonate and dilute hydrochloric acid can be measured by conducting the reaction in an open flask on a balance.



As the reaction proceeds, the calcium carbonate is consumed and converted into the carbon dioxide which escapes. The mass of the reactants therefore decreases over time.

A reading of the mass is taken every 30s. These readings are plotted in a graph of mass versus time.



The rate of reaction at any particular time can be estimated from the slope of the graph. In this case, it can be estimated by the formulae below.

$$\text{Rate of reaction} = \frac{M2 - M1}{T2 - T1}$$

- ② Estimate the overall rate of reaction from loss of reactant.

Consider the example above of estimating the rate of reaction of excess calcium carbonate and dilute hydrochloric acid.

By using a limiting amount of calcium carbonate, the end of the reaction can be observed by either noting the stopping of effervesce or the disappearance of the calcium carbonate chips added to the hydrochloric acid.

A total of 0.01g of calcium carbonate takes about 20s to complete the reaction. The rate of reaction can be calculated.

$$\begin{aligned} \text{Rate of reaction} &= \frac{\text{Amount of reactant}}{t_f - t_i} \\ &= \frac{0.01\text{g}}{20\text{s}} \\ &= 0.0005\text{g s}^{-1} \\ &= 0.5\text{mg s}^{-1} \end{aligned}$$



- ② Estimation of the rate of reaction by observations of formation of product.

To estimate the general rate of reaction, one can observe when the products formed have reached a certain mass. A reversal of the balance method mentioned above can be used and a graph of the mass versus time plotted.

Alternatively, the rate of reactions that form gaseous products can be estimated from the amount of gas formed over time.

To measure the volume of gas collected per unit time, a gas syringe can be attached to a flask fitted with a rubber stopper.

The time taken to reach the predetermined amount of gas is noted. The overall *rate of reaction* can be calculated from the formulae below:

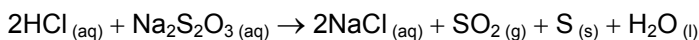
$$\text{Rate of reaction} = \frac{\text{Volume of gas collected}}{\text{Time taken to collect gas}}$$

Alternatively, the amount of gas collected is measured at 30s intervals and a graph of the volume of gas versus time is plotted.

Examples

- ① Measurement of overall rate of reaction from formation of product.

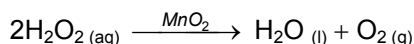
The reaction of hydrochloric acid with sodium thiosulphate results in the formation of sulphur.

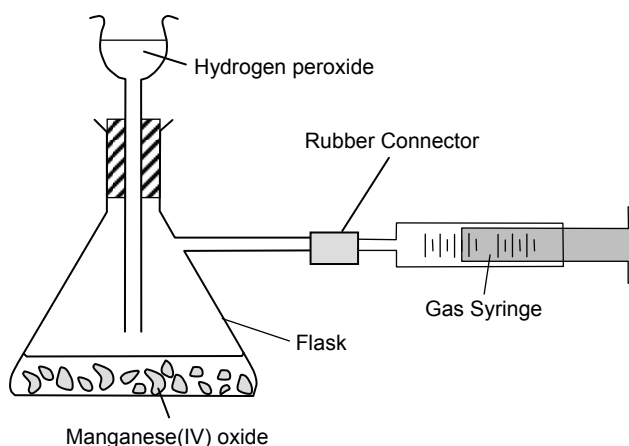


This sulphur makes the resultant solution cloudy. To measure the time taken for the reaction to occur, the reaction is carried out in a flask. The flask is first shaken when the reactants are added and a piece of paper marked with a black cross is placed under the flask. The time it takes for the solution to block out the cross is noted.

- ② Measurement of rate of reaction from formation of a gaseous product.

Hydrogen peroxide decomposes in the presence of a catalyst, manganese oxide to form oxygen and water.



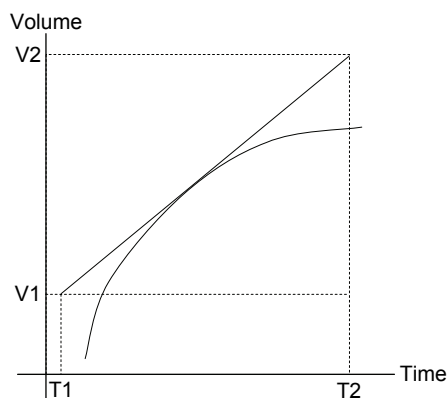


To calculate the overall rate of reaction, the time used to fill a specific volume is noted with the use of a stop watch and the rate calculated.

A total of 50 cm³ of oxygen took about 20s to collect. The overall rate of reaction thus can be calculated.

$$\begin{aligned} \text{Rate of reaction} &= \frac{\text{Volume of gas collected}}{\text{time taken to collect gas}} \\ &= \frac{50\text{cm}^3}{20\text{s}} \\ &= 2.5\text{cm}^3\text{s}^{-1} \end{aligned}$$

Alternatively, the volume of oxygen collected can be noted every 30s and a graph of volume versus time plotted. The gradient of the slope gives an estimate of the rate of reaction at any instant.



And the rate can be estimated from the formulae below.

$$\text{Rate of reaction} = \frac{V_2 - V_1}{T_2 - T_1}$$

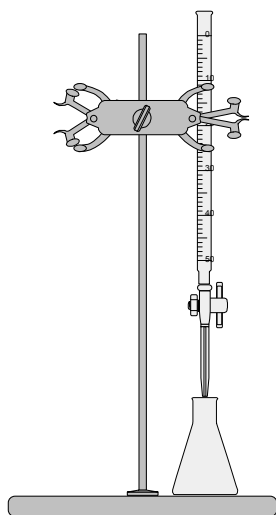


Titration

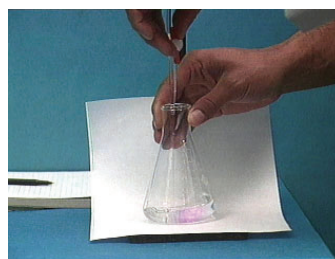
Titration is a chemical analysis to find out the unknown concentration of a solution with a solution of known concentration.

📄 Set up for titration

- The solution with known concentration is placed in the burette. This solution is also known as the *titrant*.
- A fixed volume of the solution with unknown concentration is placed in the conical flask and an indicator added to the solution in the conical flask.
- The solution in the burette is added slowly to the conical flask mixture until the end point is reached.
- Alternatively, the solution with the unknown concentration can be placed in the burette and the solution with the known concentration can be placed in the conical flask.



Set up for titration

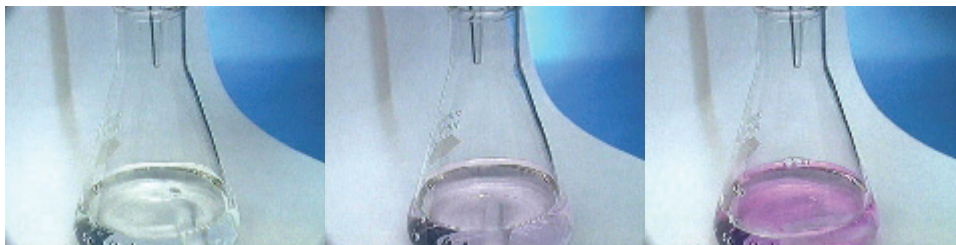


Close up of titration

📄 End point

The end point is when the reactants have almost completely reacted with each other. The end point of a chemical reaction is detected with a colour change of an indicator placed in the reaction mixture. The indicator's colour change must be within the pH range of the end point. The titration is stopped at this point.

Overshooting the endpoint of the titration by adding too much titrant is a common error. The endpoint for titration is reached when you reach a pale colour that persists throughout the solution for several seconds.



Flask **before** endpoint...

...approximately **at endpoint**...
(very faint pink tinge)

...**after** endpoint (overshot)

Indicators

Common indicators are methyl orange, phenolphthalein and universal indicator solution. Sometimes starch solutions are used for titration against iodine.



Universal Indicator solution and universal indicator paper

Calculations

After the titration, the following steps are used to calculate the concentration of the unknown solution:

- ① Write a balanced equation to show the reaction between the 2 reactants used in the titration.
- ② Determine which solution has unknown concentration. This is done by listing out all the information we have on the solutions. Remember to note the volume of solution used on the burette.
- ③ Find the number of moles of the known solution used during the reaction.
- ④ Calculate the concentration of the unknown.

Some common titration reactions are listed below:

- Titration between an acid and an alkali

An acid-base titration is usually used to find the concentration of an acid or a base.

Examples

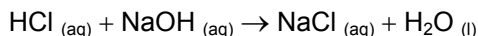
Titration of hydrochloric acid with sodium hydroxide:

- The concentration of an unknown solution of sodium hydroxide can be found using a known concentration of hydrochloric acid.

- Universal indicator solution is used as the indicator. The end point is when the indicator changes from yellow to green.

Steps

- ① Write a balanced equation to show the reaction:



From the equation, we know that 1 mole of hydrochloric acid reacts with 1 mole of sodium hydroxide.

- ② Determine which solution has unknown concentration.

Acid:	Concentration	=	0.5 mol dm ⁻³
	Volume in flask	=	0.025 dm ³
Base:	Concentration	=	unknown
	Volume used in burette	=	0.04 dm ³

- ③ Find the number of moles of the known solution used during the reaction.

$$\begin{aligned} \text{Moles of HCl used in the reaction} &= \text{Concentration} \times \text{Volume} \\ &= 0.5 \text{ mol dm}^{-3} \times 0.025 \text{ dm}^3 \\ &= 0.0125 \end{aligned}$$

From the equation, one mole of sodium hydroxide reacts with one mole of hydrochloric acid.

Therefore, no. of moles of sodium hydroxide = 0.0125

- ④ Calculate the concentration of the unknown.

$$\text{Volume of sodium hydroxide} = 0.04 \text{ dm}^3$$

$$\text{No of moles of sodium hydroxide} = 0.0125$$

Therefore,

$$\begin{aligned} \text{Concentration of sodium hydroxide} &= \frac{\text{No. of moles of sodium hydroxide}}{\text{volume of sodium hydroxide}} \\ &= \frac{0.0125}{0.04} \\ &= 0.313 \text{ mol dm}^{-3} \end{aligned}$$



- ⑤ More explanations and examples of the calculations using the mole concept are found in Chapter 3.

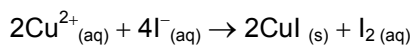
- Titrations between involving redox reactions.

Titration can be used to find the concentration of a reducing or oxidizing agent.

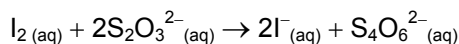
Examples

Titration of potassium iodide and sodium thiosulphate.

- The aim is to find the concentration of a solution of copper (II) ions.
- The copper (II) ions are first reacted with an excess of potassium iodide to produce iodine. In this case, the copper (II) ions act as an oxidising agent to oxidise the iodide ions to iodine:



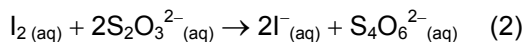
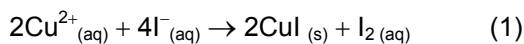
- This iodine is then titrated with sodium thiosulphate to find out the amount of iodine formed from the reaction with copper (II) ions.



- The titration is done until the colour of iodine fades (almost light yellow). Starch is then added and it forms the characteristic blue-black colour with iodine. The last few drops of sodium thiosulphate are then added till the solution turns colourless.
- After the titration, the concentration of the solution of copper ions can be found using the steps below

Steps

- ① Write balanced equations to show the reactions.



From equation (1), we know that 2 moles of copper (II) ions form 1 mole of iodine.

From equation (2), we know that 1 mole of iodine reacts with 2 moles of sodium thiosulphate.

Therefore, 1 mole of copper (II) ion produces enough iodine to react with 1 mole of sodium thiosulphate.

- ② Determine which solution has unknown concentration.

Copper (II) ions:

Concentration = unknown

Volume reacted = 0.05 dm^3

Sodium thiosulphate:

Concentration = 0.5 mol dm^{-3}

Volume used in burette = 0.021 dm^3

- ③ Find the number of moles of the known solution used during the reaction.

$$\begin{aligned}
 \text{Moles of sodium thiosulphate used in the reaction} &= \text{Concentration} \times \text{Volume} \\
 &= 0.5 \text{ mol dm}^{-3} \times 0.021 \text{ dm}^3 \\
 &= 0.0105
 \end{aligned}$$

From the equation, 1 mole of copper (II) ion produces enough iodine to react with 1 mole of sodium thiosulphate

Therefore, no. of moles of copper (II) ions = 0.0105

- ④ Calculate the concentration of the unknown.

Volume of copper (II) ions solution = 0.05 dm^3

No of moles of copper (II) ions = 0.0105

Therefore,

$$\begin{aligned}
 \text{Concentration of copper (II) ions} &= \frac{\text{No of moles of copper (II) ions}}{\text{volume of copper (II) ions}} \\
 &= \frac{0.0105}{0.05} \\
 &= 0.21 \text{ mol dm}^{-3}
 \end{aligned}$$

☺ The above procedure can work for any oxidizing agent that produces iodine from reaction with potassium iodide solution.

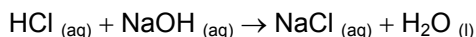


Worked Examples

Example 1

Aqueous sodium chloride can be prepared by titrating aqueous sodium hydroxide with dilute hydrochloric acid.

The equation for this reaction is shown below.



- Name the 2 pieces of apparatus used to measure accurately the volumes of the solutions in this titration.
- Name a suitable indicator for this titration. Give the expected colour change of this indicator
- Explain the meaning of the symbols (aq) and (l) in this equation.

Solution:

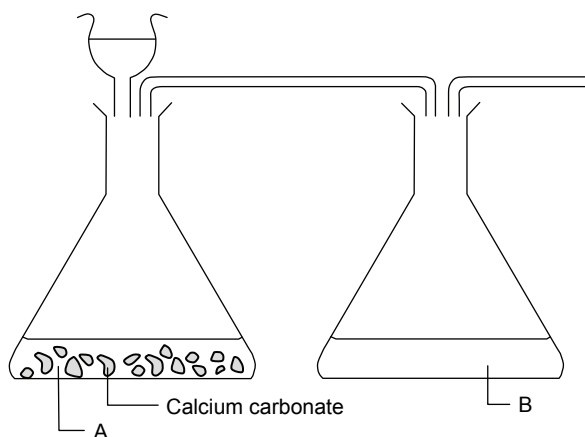
- Pipette and Burette
- Phenolphthalein or universal indicator solution. The indicator changes from yellow to green.

- c. (aq) refers to aqueous solution
(l) refers to liquid state.

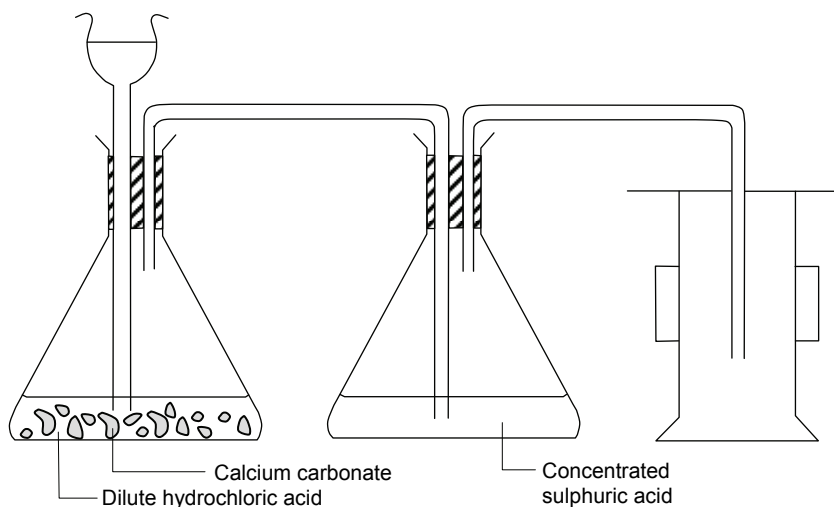


Example 2

Complete the diagram below to show the preparation, drying and collection of a sample of carbon dioxide. Name the liquids A and B.



Solution:



Since a carbonate is given, a dilute acid is added to release carbon dioxide. To dry carbon dioxide (an acidic gas), it must be passed through concentrated sulphuric acid. As carbon dioxide is denser than air, it is collected via upward displacement of air.



1.2 Methods of purification and analysis

Methods of purification

A **mixture** is made up of two or more different elements or compounds mixed together. It is possible to physically separate a mixture into its constituents.

✍ The methods of purification are used only on *mixtures*.

Examples

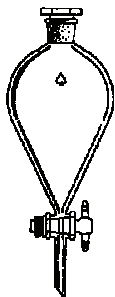
- Distillation is used to obtain pure water from salt water.
- Separation via a *separating funnel* is used to separate a mixture of oil and water.
- Fractional distillation is used to separate kerosene from petroleum
- Filtration and crystallisation are used to separate a mixture of sand and salt.



Common methods of purification

① Separation via a separating funnel

- Liquids that do not mix together are immiscible. A separating funnel can separate 2 immiscible liquids with different densities.



Separating funnel

- The mixture is placed into a separating funnel.
- It is left there till the 2 liquids separate into distinct layers. The heavier liquid is found on the bottom of the separating funnel.
- The heavier liquid is removed from the bottom of the funnel into a receiving beaker or flask.

Examples

- A mixture of oil and water can be separated using this method.
- The mixture is placed in to a separating funnel.
- Once the two distinct layers separate, the water (heavier fluid) is removed from the bottom of the funnel via the stopcock into a receiving beaker.
- The emulsion in the centre is discarded before the oil (lighter fluid) is dispensed into another receiving beaker.



2 Solvent extraction

- This method is possible when one of the substances on the mixture can dissolve in a particular solvent while the other substances present in the mixture cannot dissolve in this solvent.
- A substance can be separated based on whether it can dissolve in polar or non-polar solvents. Typical polar solvents are water and ammonia. Ionic compounds or molecules that dissociate into ions can readily dissolve in polar solvents. Non-polar solvents, such as benzene and acetone, will dissolve many organic compounds.
- The mixture is placed into excess solvent. The resultant solution is well mixed to ensure that the substance is dissolved completely.
- Filtration is used to obtain the final form of the insoluble substance.
- Crystallisation is used to obtain the final form of the soluble substance.

☺ Filtration and Crystallization will be explained in detail in the following sections.

Examples

- A mixture of naphthalene and salt can be separated using this method.
- The mixture is placed in excess water to dissolve the salt. Alternatively, benzene can be used as a solvent to dissolve the naphthalene.
- If water is used, filtration can be used to obtain the naphthalene. Alternatively, if benzene is used, filtration would yield the salt.
- To obtain the other component of the mixture in solution, crystallisation can be used.



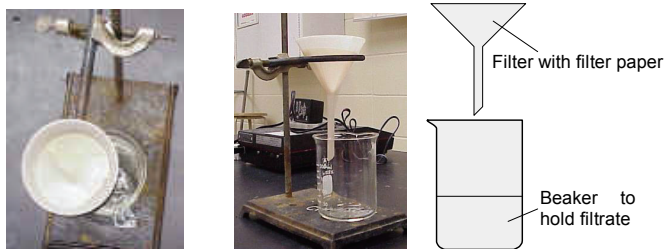
3 Filtration

- Filtration is a common and simple way of separating insoluble substances, such as a solid dissolved in a liquid. The filtrate refers to the separated liquid.
- An appropriate grade of filter paper must be used to ensure the separation is complete. Filter papers are graded according to their pore size. The pore size states the largest size of the particles that can pass through the filter.



Filter Papers

- Large volume of liquid can be filtered through a Buchner funnel with a filter pump attached. Foods such as sugar and vinegar are filtered to remove impurities.



Set up for filtration

- The filter paper is moistened before the mixture is slowly poured into the cavity. Care is taken to ensure that the level of liquid does not exceed the level of the filter paper.
- The residue in the filter paper is then washed with distilled water or deionised water and dried in an oven.

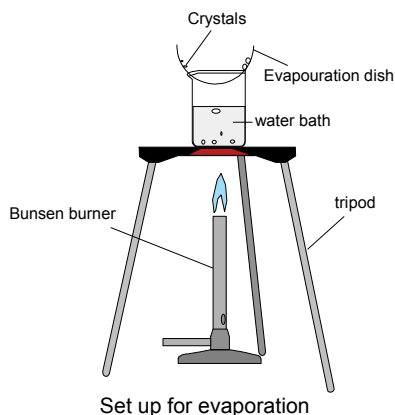
Examples

- A mixture of sand and salt (in solution) can be separated using this method.
- The mixture is placed in excess water to dissolve the salt.
- The filter paper is first moistened before the mixture is slowly added, making sure that the level of liquid does not exceed the level of the filter paper.
- The residue in the filter paper is then washed with distilled water or deionised water and dried in an oven.



4 Crystallisation

- Crystallisation refers to the formation of solid crystals from a homogenous solution. Crystallisation is often used to separate a compound from its solution. It can be used in conjunction with solvent extraction or filtration to obtain pure crystals of a compound.
- Repeating the crystallising process will improve the purity of the crystals obtained.



• Steps

- ① A saturated solution is first prepared. This is often done by dissolving excess of the compound in the solvent or by gentle heating of the solvent to remove excess solvent.
- ② It is then filtered to remove impurities and evaporated to half its volume in an evaporating dish.
- ③ The filtrate is then cooled and crystals will form.
- ④ Another filtration is done to collect the crystals, and the crystals are left to dry, usually in a desiccator.



- ☺ Note that in crystallization, the recovered compound is the solute. For example, the solute, salt is recovered from the mixture of salt in water.

Examples

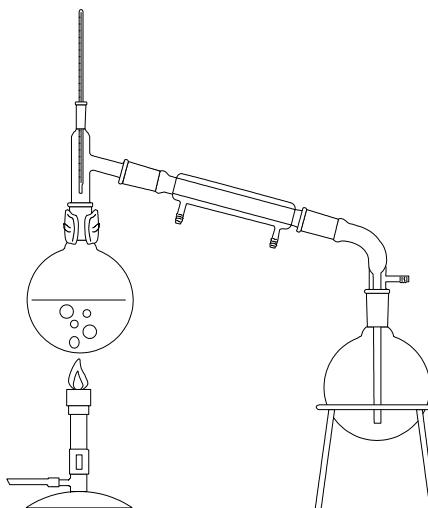
- A mixture of salt in solution can be separated using this method. Consider the previous filtrate we obtained from filtering the sand and salt mixture.
- A saturated solution is prepared by gently warming the filtrate to drive off the excess water.
- The resultant solution undergoes another filtration to remove the impurities. It is then evaporated to half its volume.
- It is then cooled and salt crystals form.

- The crystals are collected via another filtration and dried in a desiccator.



5 Simple distillation

- Distillation is used in organic chemistry for separating and purifying a liquid from a *non-volatile* solute. It involves evaporation and condensation of the liquid.



Set up for simple distillation

- Steps
 - ① The mixture is first placed in a round bottom flask and heated.
 - ② The liquid is evaporated and the vapour travels down into the condenser where it is condensed and cooled back into liquid form.
 - ③ The liquid is collected from the condenser with a conical flask. This collected liquid is called the *distillate*.
- ☺ Note that in distillation, the recovered compound is the solvent. For example, the solvent, water is recovered from the mixture of salt in water.
- ☺ When using a condenser, always remember to turn on the cooling water in the condenser before starting the actual distillation or fractional distillation process. This is to prevent formation of a vapour cloud when the process starts. Many solvent vapours are hazardous when inhaled and some can explode when a high concentration is released into air.

Examples

- A mixture of salt and water can be separated using this method.
- The mixture is placed in the round bottom flask and heated.

- The water is evaporated and the water vapour travels up to the top of the flask and passed through the condenser. The condenser condenses the water vapour back to liquid water.
- The liquid water is collected with a flask at the end of the condenser.

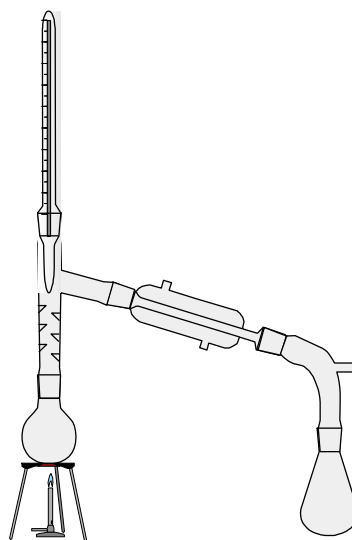
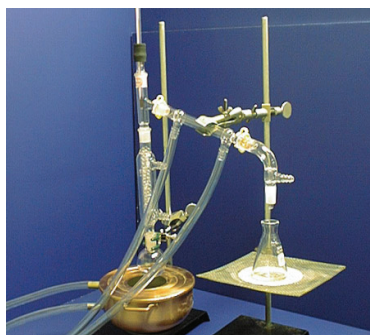


6 Fractional distillation

- Fractional distillation is one of the methods of separating and purifying liquid mixtures. It separates liquids with different boiling points.
- The main difference between the set up for simple distillation and fractional distillation is the presence of a fractionating column. These columns come in various designs and the purpose of the column is to make the mixture undergo a temperature gradient as it travels up the column.



Various distilling columns



Set up for fractional distillation

- ☺ There are some mixtures of liquids that cannot be separated via fractional distillation. These are known as azeotropic mixtures. Azeotropic mixtures evaporate to give a vapour mixture with the same composition as the liquid mixture. Azeotropic mixtures only form with a

certain composition of mixtures. For example water and benzene in the ratio of 91.1% benzene and 8.9% water forms an azeotropic mixture, which boils at a constant boiling point of 69.4°C

- ☺ In general, the longer the fractionating column, the better the separation as the mixture undergoes many more stages of evaporation and condensation. Processes like oil refining require complex calculations to optimize the length and cost of the fractionating column.

Examples

- A mixture of alcohol and water can be separated with this method.
- The mixture is placed in a round bottom flask attached to a fractionating column.
- The mixture undergoes many small vaporisations and condensation steps on the packing in the fractionating column. At each stage of vaporisation and condensation, more alcohol gets evaporated than water.
- The alcohol thus moves up to the top of the column while the water moves downwards. As it moves up, the vaporisation and condensation steps purify the alcohol as more of it becomes vapour.
- The alcohol eventually come out of the top of the column and is condensed.
- The process is used to produce alcohols with high alcoholic content. Examples are brandy from wine, whisky from fermented barley or corn, gin from fermented grains, rum from fermented molasses and vodka from fermented potatoes.



Chromatography

- ✍ Chromatographic techniques are used to separate mixtures that contain chemically similar substances.
- ✍ Quantities used are often very small. The mixture to be analysed are also known as *analytes*.
- ✍ Chromatography is used to identify many chemical and biological compounds, such as insecticides and amino acids. The purity of pharmaceutical products is also tested with chromatography.
- ✍ Chromatography is based on the theory that substances are carried through a stationary phase by a mobile phase at different speeds:
 - Chromatography involves a sample (or sample extract) being dissolved in a mobile phase (which may be a gas, a liquid or a supercritical fluid)
 - The mobile phase is then forced through an immobile, immiscible stationary phase

- The phases are chosen such that components of the sample have different solubility in each phase.
- A component which is quite soluble in the stationary phase will take longer to travel through it than a component which is not very soluble in the stationary phase but very soluble in the mobile phase.
- As a result of these differences in mobility, sample components will become separated from each other as they travel through the stationary phase.

Examples

- *Thin-layer* chromatography or *paper* chromatography can separate black ink into its components.
- Gas chromatography (GC) or *gas liquid* chromatography (GLC) can be used to detect impurities in solvents, like traces of isopropyl alcohol in ethanol.
- High performance *liquid* chromatography (HPLC) can be used to detect banned drugs in urine samples of athletes.



1 Paper chromatography

- ✍ Paper chromatography makes use of paper as a stationary phase and liquid solvent such as water or ethanol as the mobile phase.
- ✍ The procedure for making a basic paper chromatograph is discussed below:
 - ① Spots of the mixture or analytes are placed along a baseline drawn by pencil.

Pencil serves as a suitable marker as it does not dissolve in most solvents and it does not interfere with the spots.

Spots must be small. Otherwise they will interfere with other spots and become unclear

- ② The end of the paper closer to where the spots are located is dipped into a pool of solvent.

The solvent travels through the paper through capillary action.

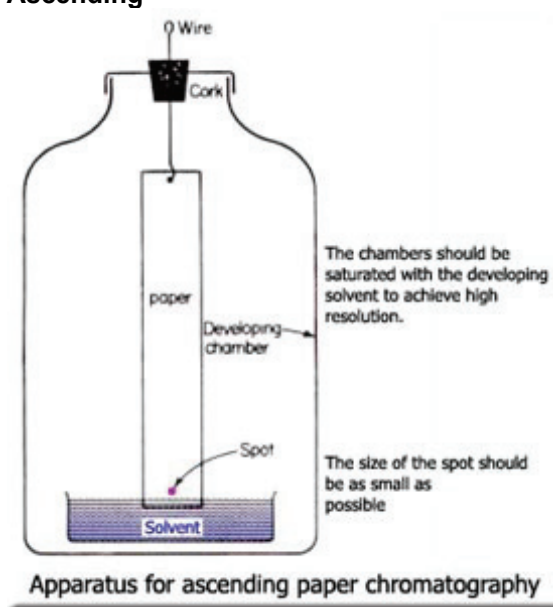
The start line should be placed above the level of the solvent. Otherwise the chromatogram will smudge and become unclear.

- ③ The setup is placed in a glass tank with a lid to prevent interference from the environment

The paper should also not touch the side of the container.

- ④ As time passes, the solvent travels to the other end of the paper. The edge of the solvent level is called the solvent front.
 - ⑤ Once the solvent front has travelled a sufficient distance, the chromatogram can be analysed. It is removed from the tank and the solvent front marked with pencil.
 - ⑥ The distance travelled by each spot and the distance travelled by the solvent front from the baseline is measured and used for analysis.
- ✍ Paper chromatograms can be developed via an ascending or descending method.

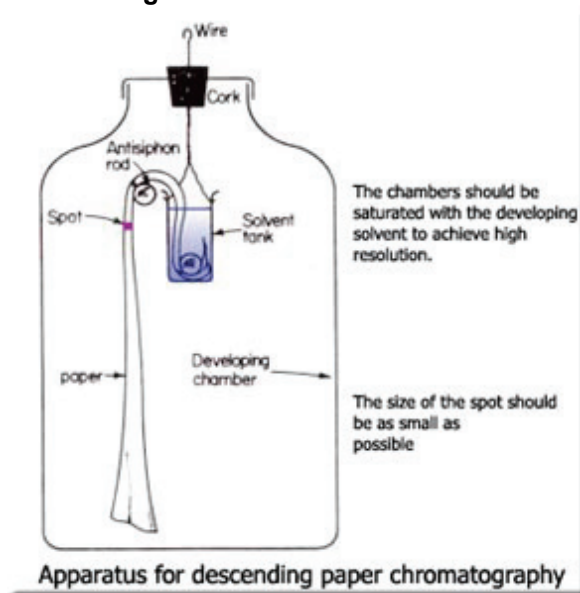
① **Ascending**



The bottom edge of the paper is in contact with the solvent and it draws the solvent upwards.

However, the solvent will rise only 20 to 25 cm and separations of the analytes are limited.

② Descending



The top of the paper is dipped in a solvent through. Capillary action and gravity draws the solvent downwards.

This technique permits a separation over a longer distance and allows an increase in resolution.

- 📄 Paper chromatograms can also be applied to colourless substances. A number of methods are available to make the separated substances visible:
 - Some colourless substances show up under UV light.
 - A chemical reagent can be sprayed onto the chromatogram to make the separated substances visible.

Examples

- The separation of Cobalt (III) ions and Nickel (III) ions in solution results in a colourless chromatogram.
- The solvent used is a mixture of acetone, water and concentrated hydrochloric acid in the ratio of 57:5:8 by volume.
- The spots are not visible in the chromatogram, but become clear when the chromatogram is sprayed with rubeanic acid. This is an example of using a chemical treatment to make the chromatogram visible.



② Other chromatographic techniques

- 📄 Thin layer Chromatography (TLC).
 - The stationary phase is a glass or plastic plate coated with a thin layer of powdered alumina or silica. The mobile phase can be any solvent.

- The sample spots are placed on the plate and dipped into a solvent
 - This technique is widely used in Organic Chemistry.
 - Its principles are similar to Paper Chromatography.
- 📖 High Performance Liquid Chromatography (HPLC).
- The stationary phase is coated on a special column. The mobile phase is usually a solvent or a mixture of solvents.
 - The sample is passed through the column by a high pressure pump, which is also used to mix the solvents.
 - As the sample passes through the column, it is separated into its components.
 - The separated components are then passed through a detector and their relative concentrations are recorded using a logger or a PC.
- 📖 Gas Liquid Chromatography (GLC) or Gas Chromatography (GC).
- The stationary phase is coated on a special column. The mobile phase is a gas like nitrogen or helium.
 - Liquid samples are usually evaporated before introduction into the column.
 - Sample is carried through the heated column by the carrier gas.
 - As the sample passes through the column, it is separated into its components.
 - The separated components are then passed through a detector and their relative concentrations are recorded using a logger or a PC.
 - This technique can be used to analyse mixtures of gases or liquids.
- 📖 Other forms of Chromatography
- Ion Chromatography (IC) uses similar principles to separate and identify the ions present in water samples.
 - Capillary Electrophoresis (CE) is used to separate and decode DNA.

Purity of substances

- 📖 A substance is deemed pure when there is only one component or compound present in the substance.
- 📖 The purity of substances can be tested with a variety of methods. A few of the more common tests are listed below:
- Melting point or boiling point determination

- Chromatography
- Density
- Specific heat capacity
- Conductivity
- Refractive Index
- thermal conductivity

✍ In particular, pure substances have fixed melting and boiling points.

Examples

Substance	Melting Point (°C)	Boiling Point (°C)	Description
Ammonia	-78	-33	Colourless gas, pungent odour
Bromine	-7	59	Reddish- brown liquid, volatile
Butane	-138	0	Gaseous hydrocarbon
Ethanol	-117	79	Colourless liquid, water-soluble
Ethanoic Acid	16	118	Colourless liquid, smells like vinegar
Methylbenzene	-95	111	Colourless organic liquid, a solvent
Propanone (Acetone)	-95	56	Colourless volatile liquid, flammable, water- soluble
Water	0	100	Colourless and odourless gas (steam), colourless liquid

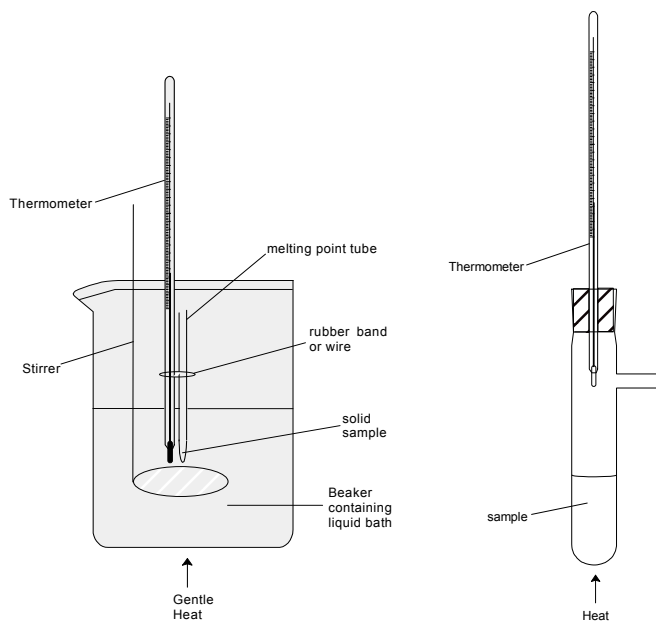
- Pure water freezes at 0°C and boils at 100°C. If some salt is added to the water, the freezing and boiling point will change. The freezing point is lowered while the boiling point rises. Saturated salt solution freezes at -22°C.



- ☺ Alloys are a mixture of metals that has a sharp melting point different from that of the individual pure metals. For example, pure lead has the melting point of 327°C and tin has the melting point of 232°C. However, an alloy made of 62% tin and 38% lead has a melting point of 183°C. the lower melting point makes this alloy useful for soldering.

Tests for purity

Determination of melting point and boiling point



Determination of melting point

Determination of boiling point

- A small amount of sample is placed in the tube.
- System is heated but not completely sealed as sealing would cause overheating and change in the pressure of the system.
- Care is taken not to heat the liquid bath or container too fast or beyond the substance's known melting point or boiling point.
- The scale of the thermometer is also carefully selected to be within the melting point or boiling point of the sample.
- Temperature is taken at first signs of melting or boiling.

Examples

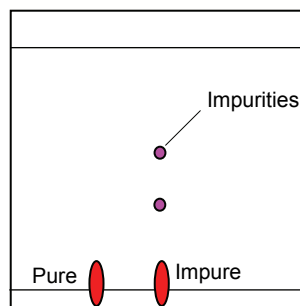
- The procedure for the determination of the melting point of naphthalene is described below:
 - ① Some naphthalene is ground up.
 - ② A small quantity is placed in a melting point tube and the tube is tapped to compact the powder to the closed end.
 - ③ When there is about 1 cm of the powder in the tube, the tube is attached to the thermometer and placed in a boiling tube containing liquid paraffin.
 - ④ The paraffin is warmed carefully with slow stirring.
 - ⑤ The temperature at which the powder rapidly shrinks and forms a clear liquid is noted. This is the melting point.

- The procedure for determining the boiling point of ethanol is described below:
 - ① Some ethanol is placed in the holder.
 - ② The sample is then heated slowly.
 - ③ Once there is a steady stream of vapour, the temperature remains constant.
 - ④ This temperature is the boiling point.




Chromatography

- Chromatography can be used to test for purity of substances.
- In paper chromatography, a pure substance will produce only 1 spot in the chromatogram. An impure substance will produce 1 intense spot which corresponds to the substance and several faint spots which correspond to the impurities present.



Importance of purity

-  Purity is important for compounds used in many fields. Some examples for the importance of purity are listed below:
 - Silicon and germanium used for the manufacturing of semiconductors, such as transistors and diodes, need to be very pure. Impurities compromise the quality of the devices.

Examples

- Silicon is used in the manufacturing of computer microchips. If the silicon used is impure, the speed of the computer would be slower than what the chip was designed for.
- In the manufacturing of drugs, it is important to know the quality of the active ingredient. If the active ingredient is impure, the amount administered on the patient might be different from what the doctor has prescribed. Impurities in the drug could also be dangerous to the patient.
- Another important area is in the manufacturing of foodstuffs. The purity of food additives should be controlled and harmful substances monitored.



Worked Examples

Example 1

Select from the following list, one method by which each of the following may be separated from the stated mixture. You may use a method once, more than once, or not at all.

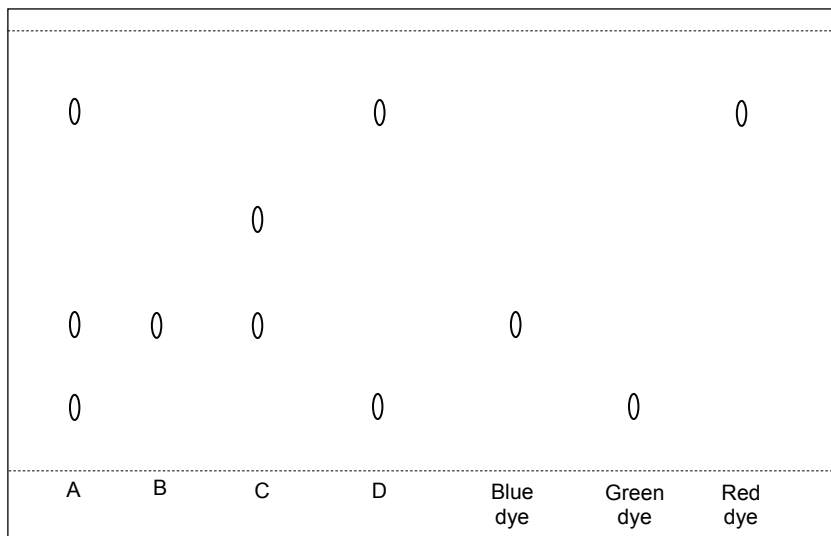
chromatography	electrolysis
precipitation	crystallisation
filtration	reduction
distillation	oxidation
sublimation	synthesis

- Red dye from a mixture of red and blue dye in solution
- Oxygen from liquid air
- Barium sulphate from a mixture of barium sulphate and water
- Ammonium chloride from a mixture of ammonium chloride and sodium chloride
- Amino acids from a mixture obtained by the hydrolysis of a protein.

Solution:

- Chromatography
- Distillation
- Filtration
- Sublimation
- Chromatography



Example 2

- a. The diagram above shows a chromatogram obtained using solutions of 3 single dyes (blue, green and red) and four other solutions (A, B, C and D).
- Which one of the solutions, A, B, C or D, contains the following?
- (i) one dye only?
 - (ii) three of the dyes?
 - (iii) green and red only?
 - (iv) a dye other than blue, green and red?
- b. In preparing the chromatogram, the following instructions were given. Suggest a reason for each instruction.
- (i) The starting line should be drawn with a pencil rather than with ink.
 - (ii) At the end of the experiment, the solvent front should be near the top of the paper
 - (iii) The spots of the solutions and dyes on the starting line should be small.

Solution:

- a. (i) B
(ii) A
(iii) D
(iv) C
- b. (i) Ink contains dye. By drawing the line with ink, it will contaminate the solvent and give inaccurate results.
(ii) This is to ensure separation
(iii) Spots should be small to prevent the spreading of the dyes sideways. This will cause the spots to mix with the spots next to them and mess up the chromatogram.



1.3 Identification of ions and gases

Tests for ions and gases

The summaries of the various tests are below:

Cations	Test	Test Results
Aluminium (Al^{3+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	White precipitate forms Precipitate soluble in excess White precipitate forms Precipitate insoluble in excess
Ammonium (NH_4^+)	Add sodium hydroxide Warm	Ammonia gas is evolved. Detect by characteristic odour, and gas turns moist red litmus blue.
Calcium (Ca^{2+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	White precipitate forms Precipitate insoluble in excess No precipitate or slight white precipitate.
Copper(II) (Cu^{2+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	Light blue precipitate forms Precipitate insoluble in excess Light blue precipitate forms Precipitate soluble in excess to give dark blue solution
Iron(II) (Fe^{2+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	Dirty green precipitate forms Precipitate insoluble in excess Dirty green precipitate forms Precipitate insoluble in excess
Iron(III) (Fe^{3+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	Reddish brown precipitate forms Precipitate insoluble in excess Reddish brown precipitate forms Precipitate insoluble in excess
Lead(II) (Pb^{2+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	White precipitate forms Precipitate soluble in excess White precipitate forms Precipitate insoluble in excess
Zinc (Zn^{2+})	Add sodium hydroxide In excess Add aqueous ammonia In excess	White precipitate forms Precipitate soluble in excess White precipitate forms Precipitate soluble in excess
Hydrogen (H^+) or acids	Moist blue litmus	Turns red

☺ Abbreviations can be used here. Precipitate is sometimes written as "ppt". solution as "soln"

☺ Although your goal is identification, experiments still must be performed with meticulous care. You must make careful observations, keep a careful record in your notebook, and perform all laboratory operations correctly. If your technique is sloppy, your identification is likely to be ambiguous or in error.

Anions	Test	Test Results
Carbonate (CO_3^{2-})	Add dilute acid	Effervesce of carbon dioxide form. Gas forms cloudy precipitate when passed through lime water
Chloride (Cl^-)	Acidify with dilute nitric acid Add aqueous silver nitrate	White precipitate forms
Iodide (I^-)	Acidify with dilute nitric acid Add aqueous silver nitrate	Yellow precipitate forms *Precipitate dissolves in hot water to form a colourless solution. On cooling, golden yellow crystals are formed
Nitrate (NO_3^-)	Add aqueous sodium hydroxide, then aluminium foil and warm carefully	Ammonia gas is evolved. Detect by characteristic odour, and gas turns moist red litmus blue.
Sulphate (SO_4^{2-})	Acidify with dilute nitric acid Add aqueous barium nitrate	White precipitate forms
Hydroxide (OH^-) or alkali	Moist red litmus	Turns blue
Bromine (Br^-)	Acidify with dilute nitric acid Add aqueous silver nitrate	Light yellow precipitate forms
Sulphite (SO_3^{2-})	Add dilute hydrochloric acid	Effervescence of sulphur dioxide form. Gas has suffocating odour and when bubble through acidified potassium dichromate, turns it from orange to green

Gases	Colour and Smell	Test
Ammonia (NH_3)	Colourless Pungent smell	Turns moist red litmus blue.
Carbon dioxide (CO_2)	Colourless Odourless	Turns lime water milky (Excess CO_2 clears the milkiness)
Chlorine (Cl_2)	Greenish- yellow Pungent smell (poisonous)	Turns moist blue litmus red, and bleaches it
Hydrogen (H_2)	Colourless Odourless	Produces "pop" sound with lighted splinter
Oxygen (O_2)	Colourless Odourless	Relights a glowing splinter
Sulphur dioxide (SO_2)	Colourless Choking smell	Turns acidified potassium dichromate from orange to green
Nitrogen dioxide (NO_2)	Reddish- brown Pungent smell (poisonous)	Turns blue litmus red
Water vapour (H_2O)	Colourless Odourless	Condenses on a cold surface. Turns white anhydrous copper sulphate blue. Turns cobalt chloride paper from blue to pink

Test for cations

- The test for cations is mainly based on the use of the hydroxide ions present in sodium hydroxide (a strong base) and ammonia (a weak base)
- The precipitate formed during cation tests are insoluble metal hydroxides.

Examples

Solution containing	Name of precipitate formed
zinc ions	zinc hydroxide
calcium ions	Calcium hydroxide.
aluminium ions	aluminium hydroxide

- $\text{Zn}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Zn}(\text{OH})_{2(\text{s})}$ white ppt
- $\text{Ca}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Ca}(\text{OH})_{2(\text{s})}$ white ppt
- $\text{Al}^{3+}_{(\text{aq})} + 3\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Al}(\text{OH})_{3(\text{s})}$ white ppt



- ☺ Ionic equations are good representations of the reactions that occur when tests are performed as they reflect only the ions that are involved in the reaction and eliminate the spectator ions (ions that are not involved). More on ionic equations will be covered in chapter 6.

- ✍ From the table, only 2 metallic ions form coloured precipitates.

Solution containing	Name of precipitate formed	Colour of precipitate formed
copper (II) ions	Copper (II) hydroxide.	Blue
Iron (II) ions	Iron (II) hydroxide.	Dirty Green
Iron (III) ions	Iron (III) hydroxide	Reddish Brown

- $\text{Cu}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Cu}(\text{OH})_{2(\text{s})}$ Blue ppt
- $\text{Fe}^{2+}_{(\text{aq})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Fe}(\text{OH})_{2(\text{s})}$ Dirty green ppt
- $\text{Fe}^{3+}_{(\text{aq})} + 3\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Fe}(\text{OH})_{3(\text{s})}$ Reddish brown ppt

- ✍ Some cations form precipitates which are soluble in excess sodium hydroxide or aqueous ammonium. Zinc (Zn^{2+}) is the only ion that is soluble in excess of both sodium and aqueous ammonia.

- Only 3 cations form precipitates which are soluble in excess sodium hydroxide. They are Pb^{2+} , Al^{3+} and Zn^{2+} .
 - $\text{Pb}(\text{OH})_{2(\text{s})} + 2\text{OH}^{-}_{(\text{aq})} \rightarrow \text{Pb}(\text{OH})^{-}_{4(\text{aq})}$ Colourless solution
 - $\text{Al}(\text{OH})_{3(\text{s})} + \text{OH}^{-}_{(\text{aq})} \rightarrow \text{Al}(\text{OH})^{-}_{4(\text{aq})}$ Colourless solution

- ③ $\text{Zn(OH)}_2(\text{s}) + 2\text{OH}^-_{(\text{aq})} \rightarrow \text{Zn(OH)}^-_{4(\text{aq})}$ Colourless solution
- Only 2 cations form precipitates which are soluble in excess aqueous ammonia. They are Cu^{2+} and Zn^{2+} .
 - ① $\text{Cu(OH)}_2(\text{s}) + 4\text{NH}_3^+_{(\text{aq})} \rightarrow \text{Cu(NH}_3)_4^{2+}_{(\text{aq})} + 2\text{OH}^-_{(\text{aq})}$
Deep blue soln
 - ② $\text{Zn(OH)}_2(\text{s}) + 4\text{NH}_3^+_{(\text{aq})} \rightarrow \text{Zn(NH}_3)_4^{2+}_{(\text{aq})} + 2\text{OH}^-_{(\text{aq})}$
Colourless soln
 - ✍ Ammonium salts do not form any precipitate. Instead it produces ammonia on warming with sodium hydroxide. Ammonia gas turns moist red litmus blue.
 - ✍ The test for hydrogen ions is the same as the test for acids. It turns blue litmus red.

Test for anions

- ✍ The tests for anions are based on characteristic behaviour of the anions with the reagents.
 - Carbonates (CO_3^{2-})
Dilute acids liberate carbon dioxide (CO_2)
$$\text{CO}_3^{2-}_{(\text{aq})} + 2\text{H}^+_{(\text{aq})} \rightarrow \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$$
 - Halide ions (Cl^- , Br^- and I^-)
The solution is first acidified by nitric acid (a catalyst). The products formed from reaction with silver nitrate are insoluble and precipitate out.
 - ① $\text{Ag}^+_{(\text{aq})} + \text{Cl}^-_{(\text{aq})} \rightarrow \text{AgCl}(\text{s})$ white ppt
 - ② $\text{Ag}^+_{(\text{aq})} + \text{Br}^-_{(\text{aq})} \rightarrow \text{AgBr}(\text{s})$ light yellow ppt
 - ③ $\text{Ag}^+_{(\text{aq})} + \text{I}^-_{(\text{aq})} \rightarrow \text{AgI}(\text{s})$ yellow ppt
 - Nitrate ion (NO_3^-)
The nitrate ions are reduced to ammonia.
 - Sulphate ion (SO_4^{2-})
The solution is first acidified by nitric acid (a catalyst). The product formed from reaction with barium nitrate is insoluble and precipitate out.
$$\text{Ba}^{2+}_{(\text{aq})} + \text{SO}_4^{2-}_{(\text{aq})} \rightarrow \text{BaSO}_4(\text{s})$$
 white ppt
 - Hydroxide ions (OH^-)
The test is the same as that for alkalis. It turns red litmus blue.
 - Sulphite ions (SO_3^{2-})
Dilute hydrochloric acid liberates sulphur dioxide (SO_2)
$$\text{SO}_3^{2-}_{(\text{aq})} + 2\text{H}^+_{(\text{aq})} \rightarrow \text{SO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$$

Test for gases

- ✎ The tests for gases are based on their characteristic colours and reactions with the reagents
- Ammonia (NH₃)
The only alkaline gas tested for. It has a characteristic odour and turns red litmus blue.
 - Carbon Dioxide (CO₂)
The gas turns limewater (weak solution of Calcium hydroxide) milky.
$$\text{CO}_2 (\text{g}) + \text{Ca}(\text{OH})_2 (\text{aq}) \rightarrow \text{CaCO}_3 (\text{s}) + \text{H}_2\text{O} (\text{l}) \quad \text{milky solution}$$
 - Chlorine (Cl₂)
Only greenish-yellow coloured gas. As it is an acidic gas, it turns blue litmus red. It is also a strong oxidising agent and will bleach the litmus (litmus paper turns white)
 - Hydrogen (H₂)
Gas reacts with oxygen in the air to form an explosive mixture. The “pop” sounds comes from a mini explosion caused by the presence of an ignition source (lighted splinter)
 - Oxygen (O₂)
The only gas that supports combustion, so it will relight a glowing splinter.
 - Sulphur Dioxide (SO₂)
The gas is a good reducing agent and will reduce the chromate (VI) ions (orange) to chromate (II) ions (green)
 - Nitrogen dioxide (NO₂)
Gas has a reddish brown colour and is acidic. It turns blue litmus red.
 - Water (H₂O) in the form of water vapour
The crystals used have different colours with and without their water of crystallisation. In the presence of water, a significant change in colour can be observed.

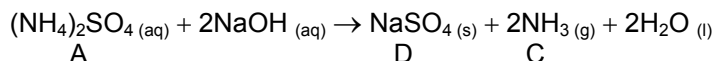
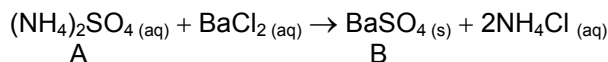
Worked Examples

Example 1

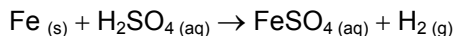
- a. Identify each of the lettered substances in the following section and write equations for each of the reactions described.
- (i) Salt A is soluble in water. Addition of barium chloride to this solution gives a white precipitate that does not dissolve in dilute hydrochloric acid. When the solution of A is warmed with sodium hydroxide solution, a gas C, which turns damp red litmus paper blue, is evolved, leaving a colourless solution D.
- b. Explain each of the following observations.
- (i) When iron reacts with dilute sulphuric acid, a pale green solution is formed and a colourless gas is evolved. (Write an equation for this reaction)
- (ii) When aqueous silver nitrate is added to hydrochloric acid, a white precipitate forms. (Write an equation for this reaction)
- (iii) On exposure to light, the white precipitate darkens.

Solution:

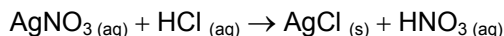
- a. (i) A is ammonium sulphate.
B is barium sulphate
C is ammonia
D is sodium sulphate solution



- b. (i) Iron reacts with dilute sulphuric acid to form iron (II) sulphate, which forms a green solution and hydrogen gas.



- (ii) Silver chloride forms as a precipitate.



- (iii) Silver chloride is affected by light. It slowly turns violet and eventually black.



Example 2

By giving the name of the formula, identify each of the lettered substances in the following accounts:

- When an excess of aqueous chlorine was added to potassium iodide, a black solid P, was formed.
- Q is a sodium salt. When a solution of Q was added to aqueous silver nitrate, a white precipitate, R was formed. This precipitate does not dissolve in dilute nitric acid.
- S is a sodium salt. When a solution of S was added to aqueous barium nitrate, a white precipitate, T, was formed. When dilute nitric acid was added to T, it dissolved and a gas, U, which turned limewater milky, was evolved.
- When aqueous sodium hydroxide was added to aqueous zinc chloride, a white precipitate V was formed. The white precipitate dissolved in an excess of sodium hydroxide to form a colourless solution in which zinc was present as a salt.

Solution:

- P is iodine, I_2
- Q is sodium chloride, $NaCl$
R is silver chloride, $AgCl$
- S is sodium carbonate, Na_2CO_3
T is barium carbonate, $BaCO_3$
U is carbon dioxide, CO_2
- V is zinc hydroxide, $Zn(OH)_2$

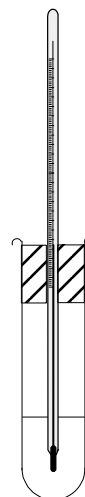
- ☺ Although barium nitrate is used as a test for sulphate, it is important to note that barium carbonate also forms a white precipitate in this test. It is the only compound that can liberate carbon dioxide. Therefore it is important to consider all the observations before concluding the identity of the ions. This also applies to practical qualitative analysis.



Worked Problems

Example 1

- a. A solid A is dissolved in a liquid, B and the following apparatus was arranged to find the boiling point of liquid B.
- Name 2 things that are wrong with the set-up and suggest corrections.
- b. Oxygen can be prepared in the laboratory by adding dilute hydrogen peroxide solutions to lumps of manganese (IV) oxide.
- (i) Write a balanced equation to describe the decomposition of hydrogen peroxide to water and oxygen gas.
- (ii) The oxygen gas can be collected using a gas syringe. Draw a diagram, with clear labels, of how you could set up this experiment.
- (iii) Describe a simple test to show that the gas is oxygen.

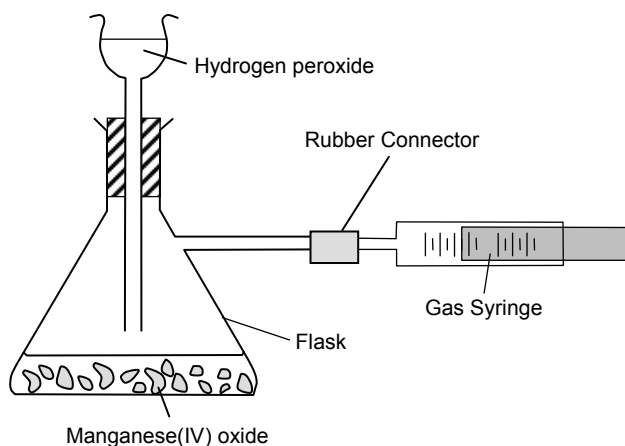


Solution:

- a. The bulb of the thermometer should not be in the liquid. The tube should not be sealed, or pressure will build up in the tube.

The thermometer should be moved out of the liquid and the stopper removed.

- b. (i) $2\text{H}_2\text{O}_2(\text{aq}) \xrightarrow{\text{MnO}_2} \text{H}_2\text{O}(\text{l}) + \text{O}_2(\text{g})$
- (ii)

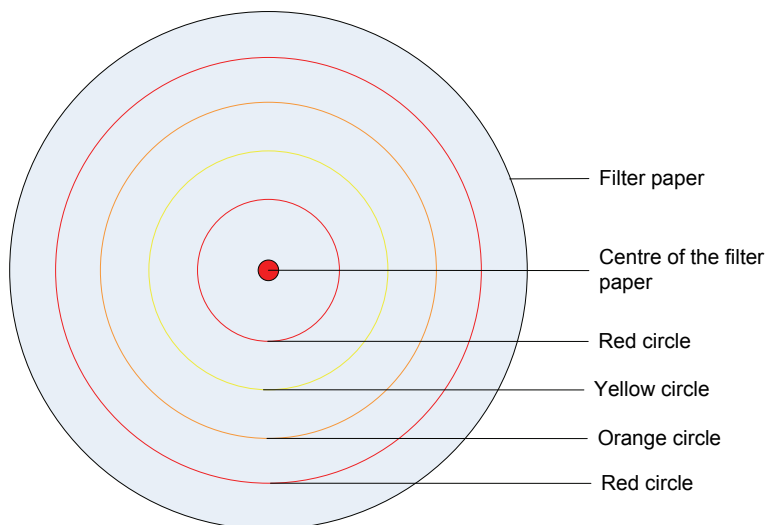


- (iii) Place a glowing splinter into the gas. It should relight.

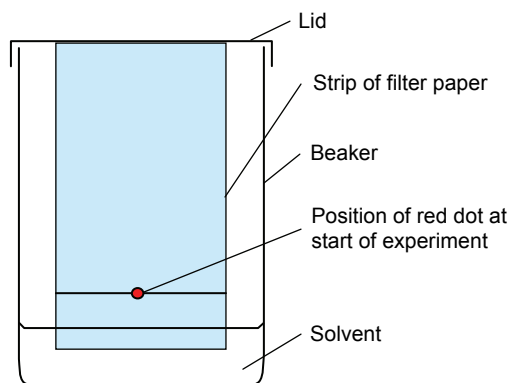


Example 2

The red colouring matter in a fizzy drink was extracted by a solvent. Two drops of the red solution were placed at the centre of a circle of filter paper and allowed to dry. Drops of pure solvent were added to the centre of the filter paper. After some time, four coloured circles were seen, as shown in the diagram below.



- a. (i) What is the name of the process described above?
(ii) Name a piece of apparatus that can be used to drop the solvent on to the filter paper.
(iii) How many different dyes are there in the fizzy drink?
- b. An alternative set of apparatus for this experiment is shown below. In this case, a strip of filter paper is used and the filter paper is dipped into a solvent.



Draw and label a diagram to show the results you would expect if this was the set up and left until the solvent reached the top of the filter paper.

Solution:

- a. (i). Chromatography
(ii) Capillary tube
(iii) There are 4 different dyes.

(b)

