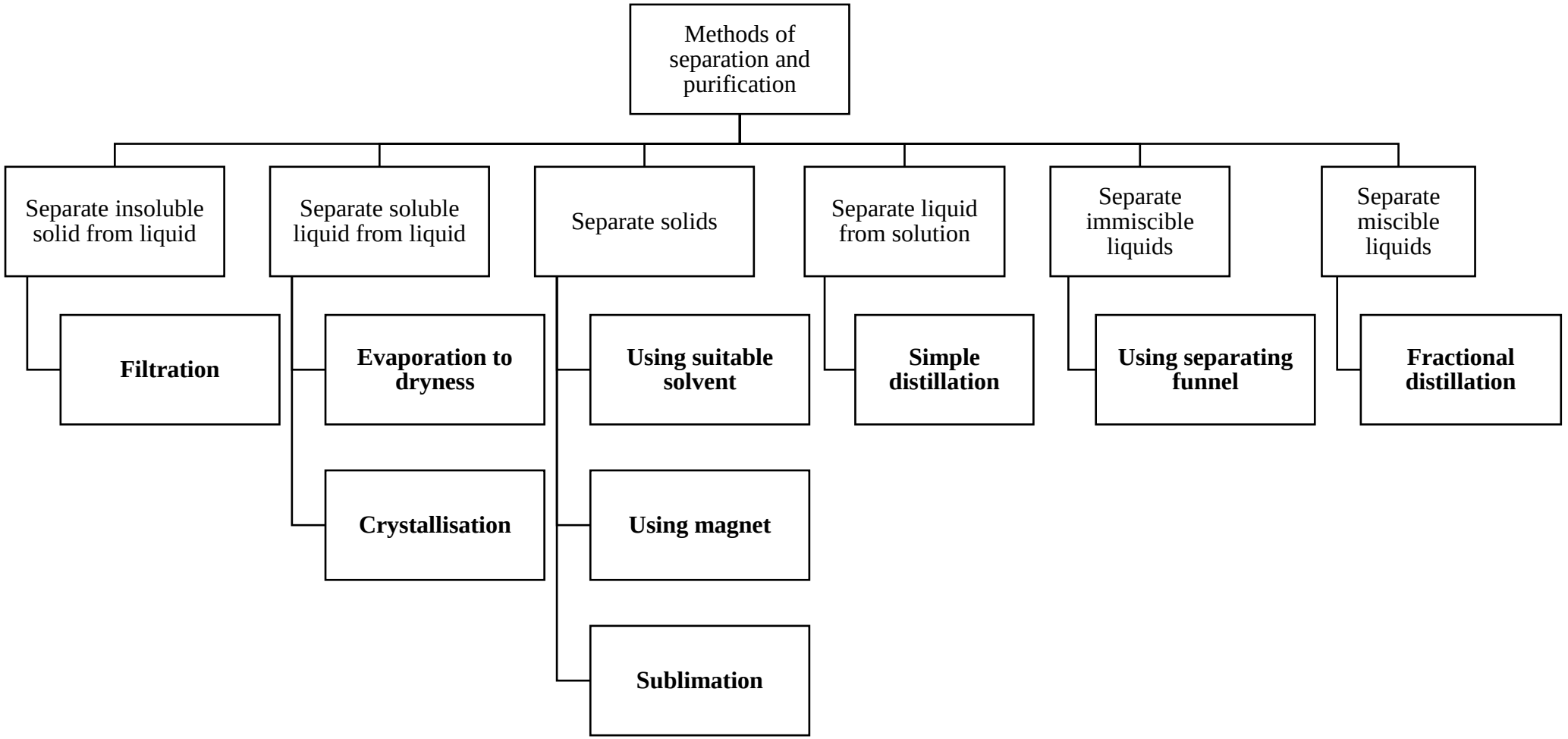
Topic 9 – Separation and Purification

Learning outcome:

1. describe methods of separation and purification for the components of mixtures, to include:
   (i) use of a suitable solvent, filtration and crystallisation or evaporation
   (ii) sublimation
   (iii) distillation and fractional distillation
   (iv) use of a separating funnel
   (v) paper chromatography
2. suggest suitable separation and purification methods, given information about the substances involved in the following types of mixtures:
   (i) solid-solid
   (ii) solid-liquid
   (iii) liquid-liquid (miscible and immiscible)
3. interpret paper chromatograms including comparison with 'known' samples and the use of $\boldsymbol{R_f}$ values
4. explain the need to use locating agents in the chromatography of colourless compounds (knowledge of specific locating agents is not required)
5. deduce from given melting point and boiling point data the identities of substances and their purity
6. explain that the measurement of purity in substances used in everyday life, e.g. foodstuffs and drugs, is important.

Separation techniques

Separation technique	Purpose	Example
1. **Filtration**	Separate insoluble solid from liquid + solid mixture	Separate sand from water + sand mixture
2. **Evaporation to dryness**	Separate soluble solid from solution	Separate salt from seawater
3. **Crystallisation**	Separate pure solid from impure solution	Separate sugar from cane syrup
4. **Sublimation**	Separate pure solid from mixture of solids	Separate iodine from iodine + sand mixture
5. **Simple distillation**	Separate pure liquid from solution containing dissolved solids	Separate pure water from impure water (seawater)
6. **Fractional distillation**	Separate miscible liquids with different boiling points	Separate different oil fractions from crude oil
7. **Separating funnel**	Separate immiscible liquids	Separate oil from water
8. **Paper chromatography**	Separate substances in a mixture based on their solubility in solvent	Separate and identify various chemicals in a drug


Methods of separation and purification
Separate insoluble solid from liquid
Separate soluble liquid from liquid
Separate solids
Separate liquid from solution
Separate immiscible liquids
Separate miscible liquids
Filtration
Evaporation to dryness
Crystallisation
Using suitable solvent
Using magnet
Sublimation
Simple distillation
Using separating funnel
Fractional distillation

3.1 Obtaining Pure Substances from Mixtures

Pure substance

made up of **one single** element / compound,
not mixed with any other substance

Mixture

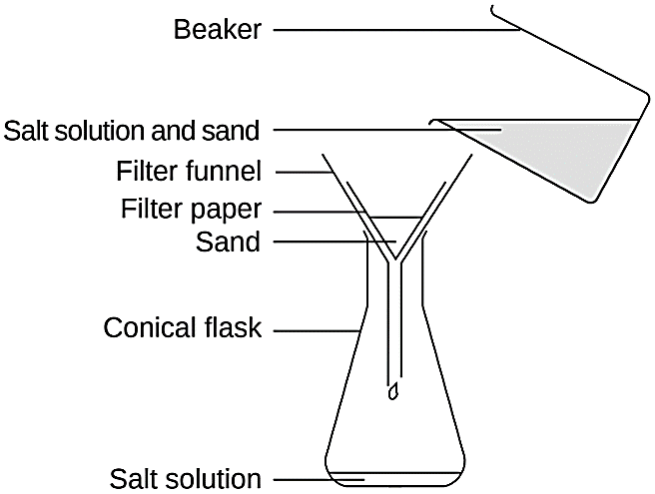
made up of **two or more** substances
that are not chemically combined

3.2 Separating a Solid from a Liquid

Filtration

Filtration

separate insoluble solid particles from liquid



Procedure

1. Pour the mixture into a filter funnel that is lined with filter paper.
2. Collect the filtrate in a conical flask.
3. Collect the residue and dry it on filter paper.

Substance	Definition	Explanation
1. **Residue**	solid remains on filter paper	large particles trapped by pores
2. **Filtrate**	liquid / solution passes through filter paper	smaller particles passed through pores

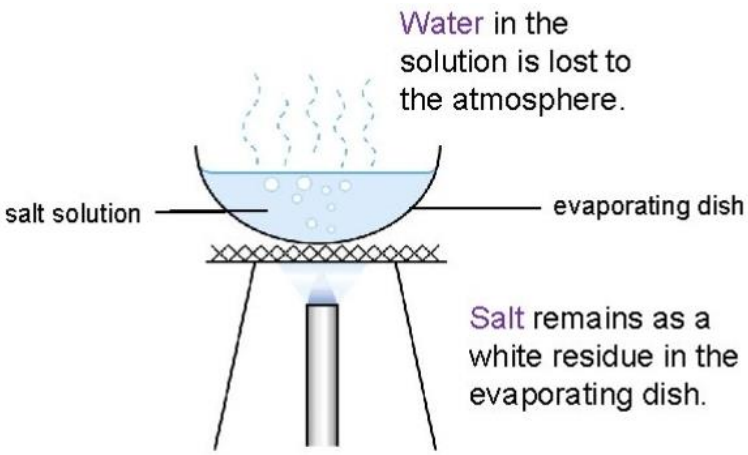
Evaporation to dryness

Evaporation to dryness

obtain soluble solid from a solution

Process: (sand + sodium chloride)

Procedure	Purpose
1. Add excess distilled water to mixture	completely dissolve sodium chloride
2. Filter the mixture	remove sand as residue
3. Evaporate salt solution (filtrate) to dryness	obtain sodium chloride



Crystallisation

Crystallisation

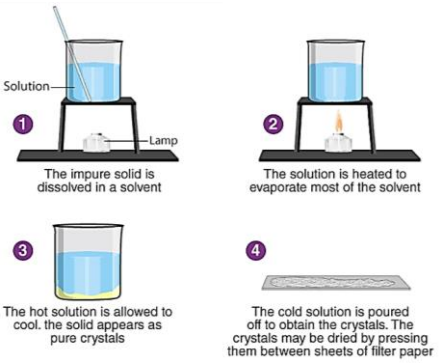
obtain a pure solid sample from its solution

Process: (glass + copper(II) sulfate)

Procedure	Purpose
1. Add excess distilled water to mixture	completely dissolve copper(II) sulfate
2. Filter the mixture	remove glass as residue
3. Heat the filtrate	obtain a saturated solution of copper(II) sulfate
4. Cool the saturated solution	solution crystallise and form crystals
5. Filter the mixture	obtain crystals, dry crystals between sheets of filter paper

Test whether a solution is saturated: dip a clean glass rod into the solution

- There will be a small amount of solution on the rod
- Small crystals form on the rod as solution cools → solution is **saturated** (at saturation point / crystallisation point)



Crystals $\xrightarrow{\text{give off water when heated}}$ powder

3.3 Separating Solids

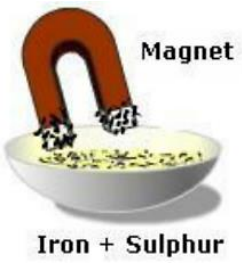
Using a suitable solvent

- To separate a mixture of two solids, the solvent:
  1. only one solid is soluble
  2. the other solid is insoluble
- Common solvents
  (a) water
  (b) ethanol

Using a magnet

Using a **magnet**

separate a magnetic substance from a non-magnetic substance

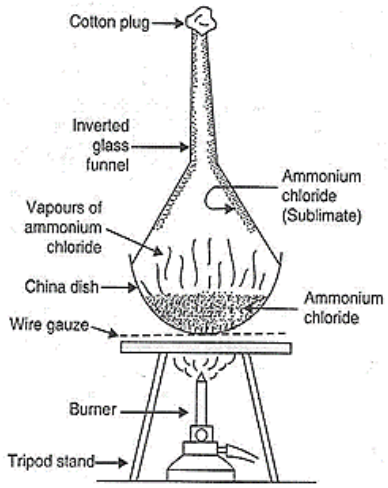


- Metals with magnetic property:
  1. Iron (Fe)
  2. Nickel (Ni)
  3. Cobalt (Co)
  4. Steel (Fe + C)
- Place the magnet above the mixture

Sublimation

Sublimation

separate a solid that sublimes from one that does not sublime



Sublime upon heating
(a) iodine (I)
(b) ammonium chloride (NH_3)

Process: (iodine + sodium chloride)

Procedure	Purpose
1. Heat the mixture	Iodine: black solid → purple vapour directly
2. Vapour condenses	changes back to solid directly on a cold surface
3. Sodium chloride	does not sublime – remains in evaporating dish

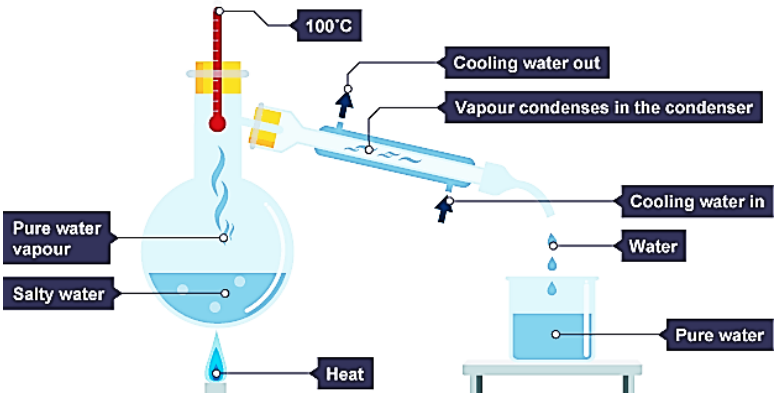
3.4 Separating a Liquid from a Solution

Simple distillation

Simple distillation

separate pure solvent (liquid) from solution

Apparatus
1. Distillation flask (solution boils)
2. Thermometer (temperature of vapour)
3. Condenser (vapour cools)
4. Receiver (collect solvent)



Physical changes
1. **Boiling** a liquid
2. **Condensing** the vapour

Procedure (salt solution)

Process	Explanation
1. In distillation flask, solution boils	Boiling chips → ensure smooth boiling (heat evenly distributed) Water vaporises, rises and enters condenser
2. In condenser, water vapour cools	Vapour condenses → pure water
3. Pure water collected	as distillate the receiver (conical flask)
4. Solution in distillation flask becomes more concentrated as distillation continues	If distillation is allowed to carry on, a solid residue of salt will be left in the flask.

Procedures to note

Apparatus	Procedure to note	Reason
Thermometer	**Bulb placed beside side arm** of distillation flask (should not be dipped into solution)	Measures boiling point of distilled substance
Condenser	• **Slope downwards**	• Pure solvent formed runs downwards → receiver (a conical flask)

	• Two tubes: inner tube + outer water jacket. Cold running water – water jacket 1. **enter from bottom** 2. **leave from top**	• If water enters from top → exits condenser before water jacket completely filled Water enters from bottom → entire jacket always <u>completely filled</u> (more efficient cooling system)
Receiver	Put into large container filled with **ice** if distillate is volatile	Keep <u>temperature of distillate low</u> → remains in <u>liquid</u> state

Graph of temperature change:

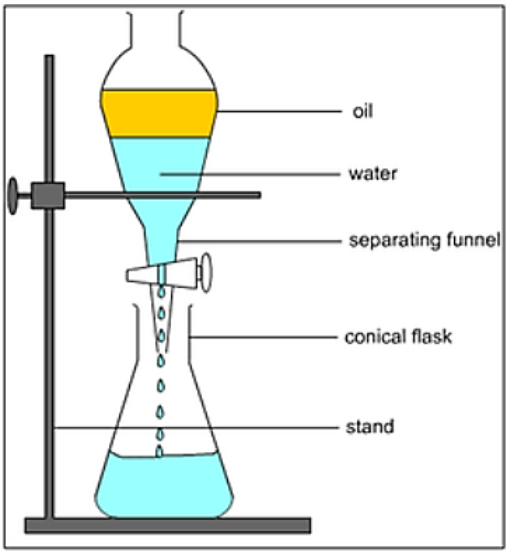
- As solution is heated, temperature increases
- When solution finally boils, thermometer records temperature of steam (100℃)
- Temperature remains unchanged until all water boiled off

3.5 Separating Liquids

<u>Using a separating funnel</u>

Separating funnel

separate immiscible liquids



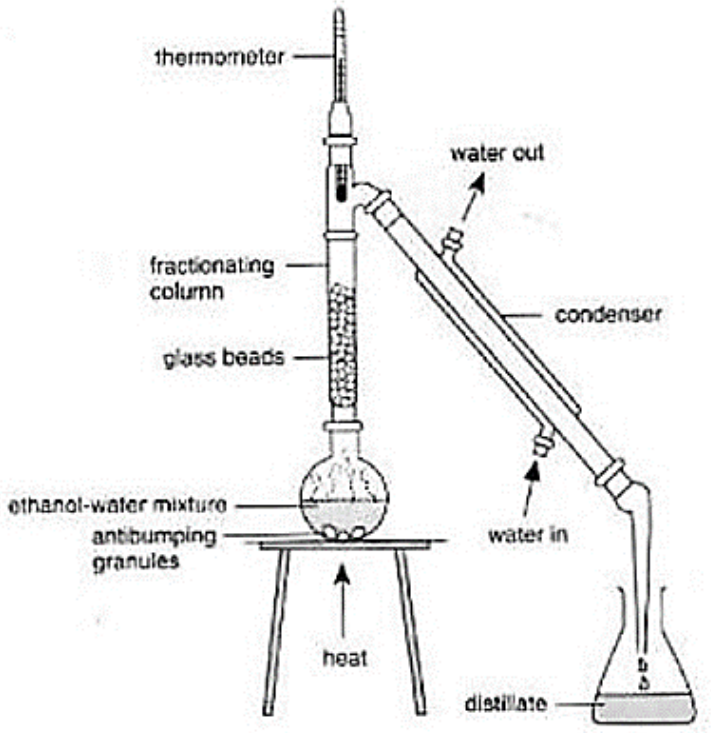
Procedure (mixture of oil + water)

1. Pour the mixture into the separating funnel (tap closed)
2. Support separating funnel using retort stand. Place clean beaker below separating funnel.
3. Allow liquids to separate completely (denser liquid at bottom)
4. Open tap of the funnel → bottom layer drain into beaker. Close tap before top layer runs out.
5. Place another beaker below the funnel. Open the tap to allow a little of the top layer of liquid into the beaker and dispose it. Now the separating funnel contains only oil; the beaker contains only water.

<u>Fractional distillation</u>

Fractional distillation

separate a mixture of miscible liquids with different boiling points



Fractionating column: surface for continuous evaporation & condensation of vapours

- Packed with glass beads
  → increase surface area for
  1. **evaporation**
  2. **condensation**
- When flask is heated, mixture of vapour from two liquids evaporate up the column

Procedure (water + ethanol)

Process	Explanation	
1. Solution is heated	Ethanol vapour + water vapour rise up fractionating column	
2. Fractionating column (thermometer shows constant temperature of 78°C – boiling point of ethanol)	Water vapour (high *bp*)	• condense in fractionating column • fall back into distillation flask
	Ethanol vapour (low *bp*)	• reach upper part of fractionating column • distil over
3. Condenser	hot ethanol vapour	condense as running water cools it
	liquid ethanol	flows down inner tube of condenser → receiver
4. Ethanol collected	as distillate in receiver	
5. All ethanol distilled over	• Temperature rises rapidly → 100°C (boiling point of water) • Water distils over, collect it separately	

Change of state

Substance	Physical change	Process
Liquid (low *bp*)	**Evaporate**	Distil over into condenser
Vapour of liquid (high *bp*)	**Condense**	Fall back into round-bottomed flask

Precautions

Measure	Explanation
1. Bulb of thermometer: positioned at opening of side arm	Accurately measure temperature of vapour passing into condenser
2. Cooling water enter the condenser jacket through lower tube & leave by upper tube	Ensure water at lower temp. than vapours – complete condensation of vapours take place more efficiently

Graph of temperature change:

- As solution is heated, temperature increases
- Solution boils → thermometer records temperature of steam (100°C)
- Temperature remains unchanged until all water boils off

Industrial applications (different boiling points)

1. Obtain nitrogen, argon and oxygen from air
2. Separate petroleum into useful fractions
3. Obtain ethanol produced by fermentation of glucose solution

Typical problems

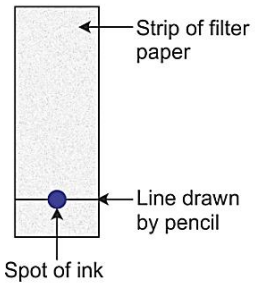
Problem	Typical problems	Solution
1. Distillation result is poor: fractions obtained are not pure.	• Distillation is carried out too quickly, components need time to separate • We need many evaporation-condensation cycles for good separation • If we supply too much heat energy to the system, less volatile components have enough energy to keep evaporating. The vapor phase is therefore not enriched with the more volatile component.	Certain amount of time is also required. Allowing the mixture to gently reflux for a while (30 minutes) before gradually increasing the energy supplied to the system through heating is typically a good strategy.
2. Collect distillate but temperature reading does not correspond to boiling point. Temperature reading is much lower.	• Thermometer is not of high quality. It should be calibrated by reading boiling distilled water. • location of thermometer bulb is essential. If it is too high, vapor condenses before thermometer can read the temperature.	The bulb should rest right above the lowest part of the adapter
3. Even though liquid in the still pot is boiling, no distillate is being collected.	• Insulation: have a look at the entire apparatus and see where the vapor has reached, by looking for drops of condensation. Insulating the top of the still pot, as well as the column and top of the three-way adapter can be beneficial. • Vapour must heat the glassware to the boiling point of the condensing liquid before it can evaporate again and rise through the apparatus. Because the distillation is performed in a fume-hood, constant air-flow inside the hood cools the apparatus.	
4. Nothing distils but amount of liquid in the still pot decreases.	• Leaks in the system. Vapour escapes though openings between joints.	Make sure all joints are properly sealed.

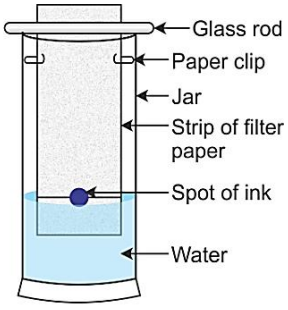
3.6 Chromatography

Paper chromatography

Procedure

1. Draw a line (starting line) with pencil near the bottom of filter paper / chromatography paper
2. Put a drop of food colouring on pencil line. Allow it to dry.
3. Dip the paper into a glass tank containing the solvent, in this case, ethanol. Ethanol that is soaked up by the paper will dissolve the dyes. (pencil line should be above the solvent level, or else the dye would wash out into the solvent)
4. Leave apparatus to stand for a while. Ethanol travels up the paper, carrying the dyes along. The more soluble a dye is in ethanol, the further it will move up the paper.





Things to take note:

- Use **pencil** to draw the start line. The sample will be dotted onto the start line using a capillary tube. A pencil is used because the pencil line will not dissolve in the solvent or be separated together with the sample. The ink from a pen dissolves and interferes with the samples.
- The beaker is **covered with a lid** during the experiment to prevent evaporation of the solvent from the beaker and the paper. Volatile solvents (ethanol, acetone) evaporate quickly.
- **Start line must be higher than solvent level**. If it is lower, sample dissolves in solvent before chromatography begins.
- Chosen **solvent must be able to dissolve sample**
  → change it if sample still remains on start line (insoluble in solvent)

Solvent front: position reached by the solvent
Chromatogram: chromatography paper with separated components

***R_f* values**
Retention factor (affected by **solubility** of substance in **solvent**)

Ratio between the distance travelled by the substance and the distance travelled by the solvent is a constant

Calculation:

$$R_f = \frac{\text{distance travelled by the substance}}{\text{distance travelled by the solvent}}$$

R_f value of substance does not change under same conditions (same solvent and same temperature)
→ easily identify a substance on chromatogram

Analysis of a sample – identify components present
Analyse a sample to
Government bodies → analyse samples of food colouring (ensure dyes used are safe for consumption)

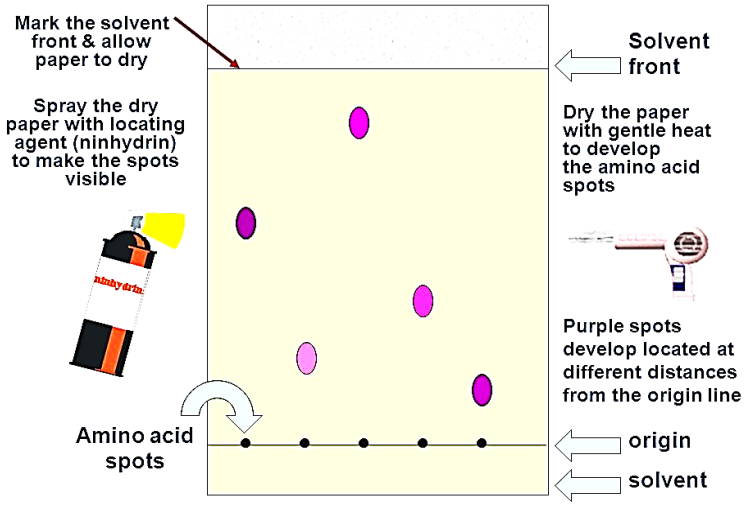
Identifying banned substance present in food colouring

1. Paper chromatography – separate the dyes in the sample.
2. Identify each dye by comparing:
   (a) its position in chromatogram with a known dye on same chromatogram
   (b) its R_f value with the R_f value of a known dye

Colourless substances
Spray **locating agent** on chromatogram → colourless substances show up as coloured spots

Steps

1. Separate mixture of amino acids by chromatography using suitable solvent
2. Stop chromatography before solvent reaches top of paper. Dry the paper
3. Spray a locating agent (ninhydrin) onto the paper
4. Locating agent **reacts** with amino acids → **coloured spots** on paper. Check R_f value of each coloured spot to identify the different amino acids



Uses

Usages of chromatography

1. **Separate components** in a sample (dyes in ink, pigments in plants, amino acids in proteins)
2. **Identify components** present in sample (traces of banned substances in food)
3. **Identify substances** (poisons, pesticides, drugs)
4. Determine **purity** of sample (pure / impure)

Ninhydrin: react with amino acids → deep blue / purple colouration (detect fingerprints)

3.7 Determining Purity

Importance of purity

Importance	Example
1. Detect **harmful impurities**	• Impurities in drugs & medicines → undesirable side effects • Chemicals (preservatives, dyes) → make food last longer / taste better / look attractive (safe for consumption)
2. Products meet **quality standards**	Production of silicon chips for electronic industry → small amount of impurities make component in electronic device less efficient

Ways to determine purity

1. **Melting point**
2. **Boiling point**
3. Chromatography

Physical properties	Impurities	Pure substance	Impure substance
1. Melting point (solid)	decrease	exact and constant	over a range of temperatures
2. Boiling point (liquid)	increase		

Chromatography carries out if:

(a) substance cannot be melted / boiled easily (non-volatile)
(b) amount of substance is very small

Purity	Chromatogram
Pure	A **single** spot (always)
Impure	More than one spot

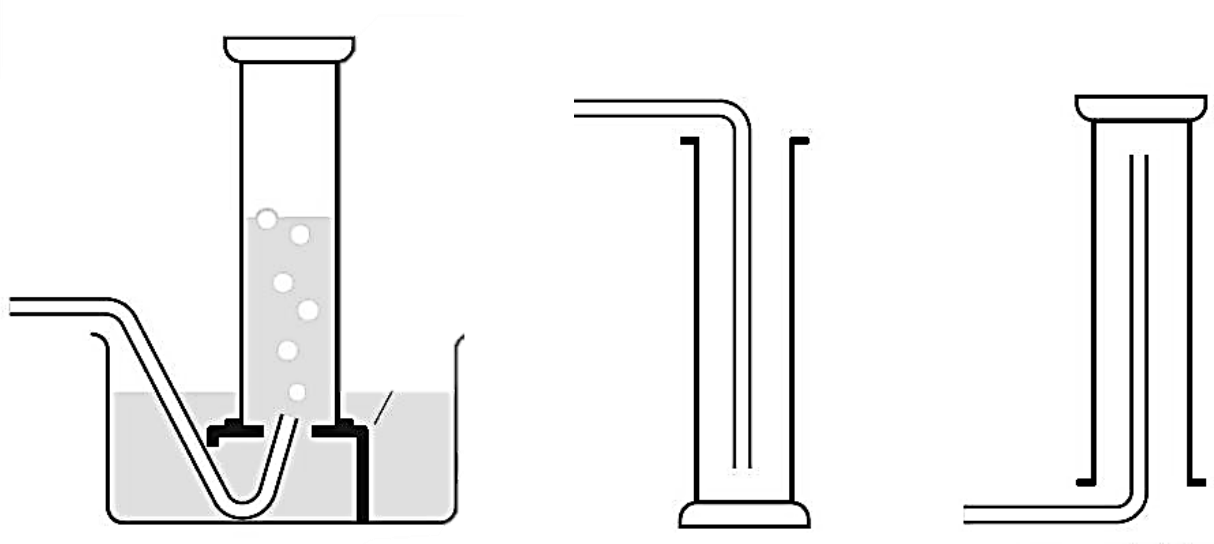
3.8 Select Suitable Apparatus for Experiments

Methods for collecting gases

Depends on the physical properties of the gas:

1. **solubility** – in water
2. **density** – compared to air

Method	Physical property		Examples
	Solubility	Density	
1. **Displacement of water**	insoluble / slightly soluble		(a) carbon dioxide (b) hydrogen (c) oxygen
2. **Downward delivery**	soluble	denser	(a) chlorine (b) hydrogen chloride (collect poisonous gases in a fume cupboard)
3. **Upward delivery**	soluble	less dense	(a) ammonia

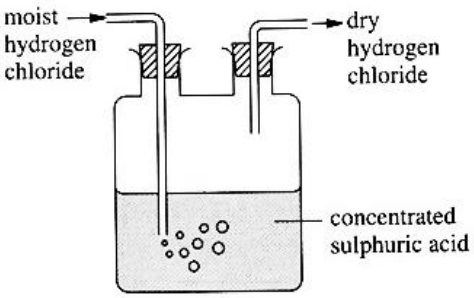


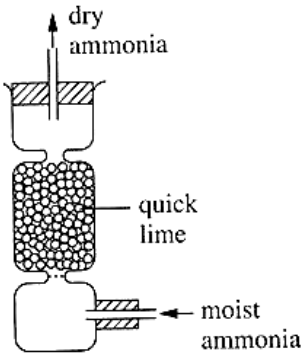
Some gases:

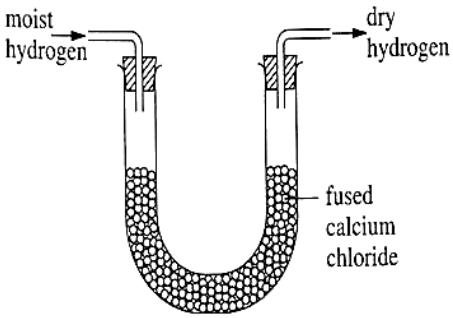
Gas	Solubility	Density	Method of collection
(a) hydrogen	not soluble	less dense	Displacement of water
(b) oxygen	very slightly soluble	slightly denser	
(c) carbon dioxide	slightly soluble	denser	
(d) chlorine	soluble	denser	Downward delivery
(e) hydrogen chloride	very soluble	denser	
(f) sulfur dioxide	very soluble	denser	
(g) ammonia	extremely soluble	less sense	Upward delivery

Dry gases – removing water

Drying agent	Dry	Examples
1. **Concentrated sulfuric acid**	Most gases	(a) chlorine (b) hydrogen chlorine × ammonia (neutralisation reaction)
2. **Quicklime** (calcium oxide)	Ammonia	
3. **Fused calcium chloride**	Most gases	







Typical questions

Separating technique

1. Paul accidentally spilled some nickel nails into a solid mixture of ammonium chloride and sodium chloride. Some properties of these substances are listed in the table below.

Substance	Thermal stability	Solubility in water	Attracted to magnet
nickel nails	heat stable	insoluble	yes
ammonium chloride	sublimes when heated	soluble	no
sodium chloride	heat stable	soluble	no

Describe and explain what Paul should do in order to obtain two substances, dry nickel nails and pure, dry sodium chloride from the mixture separately.

1 Add excess water to the mixture, and stir well to completely dissolve the ammonium chloride and sodium chloride.
2 Filter the mixture to obtain the residue (nickel nails). Dry the nickel nails between filter paper.
3 Heat the mixture in an evaporating dish. Ammonium chloride will sublime, leaving sodium chloride in the evaporating dish.

(Separation techniques used: filtration + sublimation)