Dunman High School
2022 Year 3 Chemistry
Separation Techniques

Learning Objectives

Students would 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) 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

c) 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)

d) interpret paper chromatograms including comparison with 'known' samples and the use of R_f values

e) explain the need to use locating agents in the chromatography of colourless compounds

f) deduce from the given melting point and boiling point the identities of substances and their purity

g) explain that the measurement of purity in substances used in everyday life, e.g. foodstuffs and drugs, is important

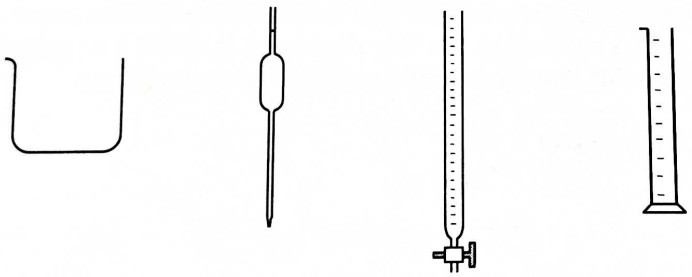
1 Measurements in Chemistry

Measurement of	Apparatus
Time	Stopwatch
Temperature	Thermometer
Mass	Electronic balance
An approximate volume of liquid	Beaker (measures an approximate volume e.g. about 50 cm^3)
An accurate volume of liquid	Measuring cylinder (measures volume to the nearest 0.5 cm^3, e.g. 22.0 cm^3)

Measurement of	Apparatus
A very accurate volume of liquid	Pipette (measures a <u>fixed</u> volume such as 20.0 cm^3 or 25.0 cm^3)  Burette (measures <u>variable</u> volumes to the nearest 0.05 cm^3, e.g. 20.05 cm^3) 
Volume of gas	Gas syringe

<u>Quick Check</u>

Which apparatus would be most suitable for measuring out 28.3 cm^3 of water to place into a flask?



A B C D (C)

[Ans: C]

Pipette (B) and burette (C) can measure volume more accurately than beaker (A) and measuring cylinder (D). Burette can measure a range of volumes while pipette can only measure a fixed volume (e.g. 25.0 cm^3).

2 Separation Techniques

- Separation techniques involve physical methods to separate mixtures into their components.

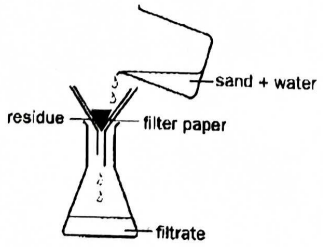
Mixture		Method of separation
Solid-Liquid	Insoluble solid + liquid	**Filtration**
	Solution (solid dissolved in liquid)	To obtain the solid (solute): **Crystallisation or Evaporation to dryness**
		To obtain the liquid (solvent): **Distillation**
Solid-Solid	One solid is soluble in a suitable solvent and one does not	Use a suitable **solvent** followed by **Filtration**
	One solid sublimes and one does not	**Sublimation**
Liquid-Liquid	Liquids that are miscible (liquids that dissolve in each other)	**Fractional distillation**
	Liquids that are immiscible	**Using separating funnel**
Various components such as dyes dissolved in the same solvent		**Chromatography**

2.1 Filtration

Use : To separate an insoluble solid from a liquid

Method : 1. Pour the mixture carefully into the filter funnel lined with filter paper.
2. The solid will be retained by the filter paper and is called the residue.
The liquid passing through the filter paper is called the filtrate.

Example : To separate sand from water



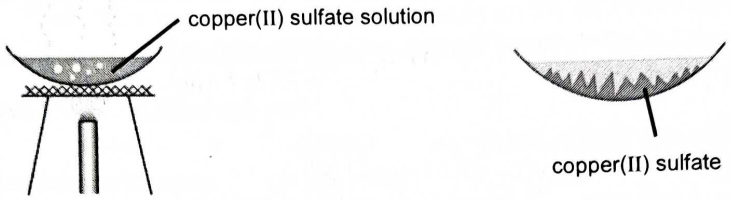
Application : Used to remove solid impurities from drinking water/chalk from salt solution

2.2 Crystallisation

Use : To obtain a crystalline substance from its solution

Method : 1. Heat the solution gently to evaporate most of the solvent so as to obtain a saturated solution.
2. Allow the saturated solution to cool. Crystals will form. This is because the solubility of a solute usually decreases when the temperature decreases.
3. Filter the mixture to obtain the crystals as residue.
4. Dry the crystals between pieces of filter papers.

Example : To obtain copper(II) sulfate from its solution

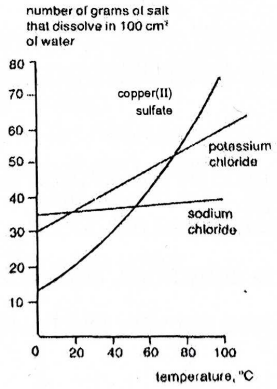


Application : White sugar (sucrose) is crystallised from sugar cane/sugar beet juices.

Solubility Curve

- A saturated solution is a solution that contains the maximum amount of solute that can dissolve in it at a given temperature.
- The maximum mass of solute that can dissolve in 100 cm^3 of water is the solubility at that given temperature.
- The change in solubility of solute in water with temperature is called the solubility curve.

The solubility curves for three salts are given in the following diagram:



Q1 The solubility curves of copper(II) sulfate and potassium chloride show that the solubility of these two salt decreases a lot on cooling, so large amount of these salts crystallise on cooling their saturated solution. Can you say the same for sodium chloride?

No, the solubility curve for sodium chloride shows that the solubility of sodium chloride remains almost the same on cooling. Little or no solid forms when a hot solution of sodium chloride is cooled.

Q2 Suggest how you would obtain sodium chloride crystal from its solution?

Evaporate the sodium chloride solution to dryness.

Q3 Explain why copper(II) sulfate crystal cannot be obtained from its solution by the method suggested in **Q2**?

If copper(II) sulfate solution is evaporated to dryness, a white powder of anhydrous copper(II) sulfate ($CuSO_4$) will be obtained instead of the blue crystal of hydrated copper(II) sulfate($CuSO_4.5H_2O$).

Note:

- A hydrated salt is a salt that contains water of crystallisation (water chemically combined with the salt in a definite proportion).
  E.g. $CuSO_4.5H_2O$, $Na_2CO_3.10H_2O$
- An anhydrous salt is a salt that does not contain water of crystallisation.
- When gently heated, hydrated salts lose their water of crystallisation to form anhydrous salt.

Example: $CuSO_4.5H_2O \rightarrow CuSO_4 + 5H_2O$

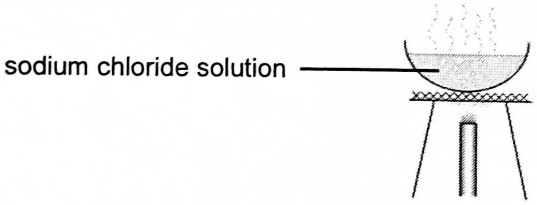
hydrated (blue crystal) — anhydrous (white powder) — water of crystallisation

2.3 Evaporation to dryness

Use : To obtain a solute from its solution

Method : Heat the solution until all the solvent has boiled off.

Example : To obtain sodium chloride from its solution



- A solute is obtained from a solution by evaporation to dryness when
  - the solute is stable to heat (does not decompose on heating)
  - solute crystal required is anhydrous, does not contain water of crystallisation
  - the solubility of solute does not vary much with temperature. E.g. NaCl
- The solid obtained by evaporation to dryness is not always pure. Any soluble impurity will be left together with the solid after evaporation of the solvent.

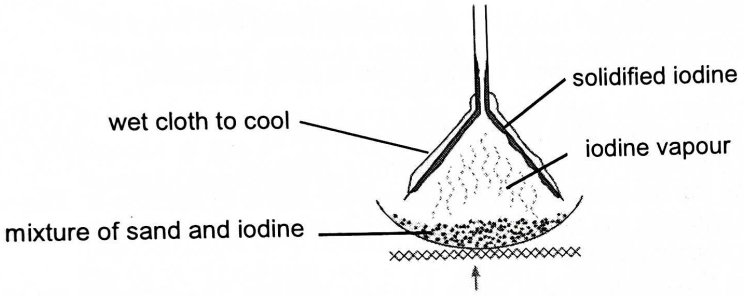
2.4 Sublimation

- Sublimation is a process in which a solid changes directly into a gas without going through the liquid state
- Substances such as dry ice (solid carbon dioxide), naphthalene (mothball), iodine and ammonium chloride can sublime.

Use : To separate a solid that can sublime from those that cannot.

Method : 1. Place an inverted filter funnel over the mixture in an evaporating dish.
2. Heat the mixture. The solid that sublimes will change from solid to vapour directly.
3. The vapour changes back to solid directly on the cool inner surface of the funnel.
4. The other substance which does not sublime remains in the evaporating dish.

Example : To separate iodine from sand

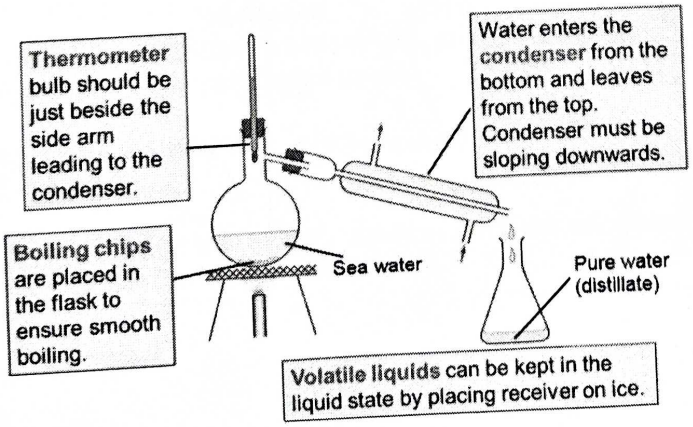


2.5 Simple Distillation

Use : To recover a solvent from a solution through evaporation followed by condensation.

Method : 1. Set up the apparatus for simple distillation.
2. A few pieces of boiling chips/boiling stones/anti-bumping granules are added to ensure smooth boiling.
3. Heat the mixture in the distillation flask. The liquid will become vapour.
4. The vapour is cooled and condensed into a pure liquid (distillate) in the condenser and is collected in a receiver (e.g. conical flask)

Example : To obtain pure water from sea water.



Q1 Why is the bulb of the thermometer placed beside the side arm of the distillation flask leading to the condenser?

This is to measure the temperature of the vapour that is distilling over. This gives the boiling point of the liquid (distillate) collected.

Q2 Why should the cooling water enter the condenser jacket through the lower tube and leave by the upper tube?

This is to ensure efficient cooling and complete condensation of vapour.

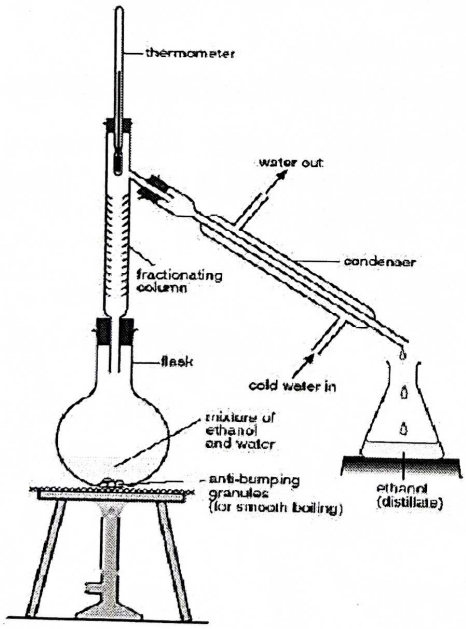
2.6 Fractional Distillation

Use : To separate 2 or more miscible liquids with different boiling points using a fractionating column.

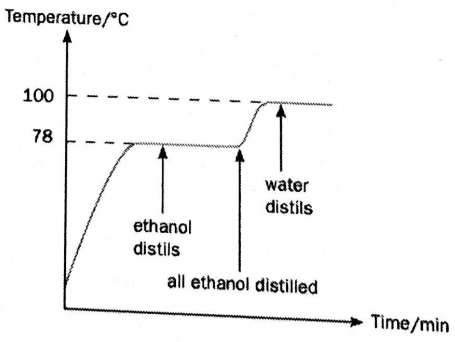
- Miscible liquids are liquids that dissolve in each other to form a solution.

Method :
1. Set up the apparatus for fractional distillation.
2. Heat the mixture in the round-bottomed flask.
3. Check the temperature when the vapour starts to distil over. The vapour of the liquid with the lowest boiling point is distilling over first. The temperature will remain constant.
4. When the temperature starts to rise, change the receiver as a different component is now distilling over.
5. Repeat step 4 until distillation is completed.

Example : To separate ethanol (boiling point 78°C) from water (boiling point 100°C)

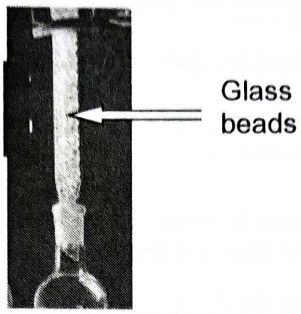


- Ethanol, having the lower boiling point, reaches the upper part of the column first and is distilled over and collected as distillate.
- The thermometer shows a constant temperature of 78°C until all the ethanol has distilled over.
- Water will distil over when the temperature reaches 100°C.



How does a fractionating column work?

- In the fractionating column, there are glass beads/helixes that provide a large surface area for vapour to condense on.



- When the flask containing two miscible liquids (e.g. a mixture containing ethanol and water) is heated, a mixture of vapours is obtained.
- The liquid with lower boiling point (ethanol) is more volatile, it evaporates more easily. The mixture of vapours contains a higher proportion of ethanol.
- In the fractionating column, the vapours start to condense on the cold glass beads. Vapour of the liquid with the higher boiling point (water) condenses more readily. Some of the liquids formed flow back into the distilling flask.
- Liquids condensed on the glass beads are evaporated by the rising hot vapours from the flask. The liquid with lower boiling point (ethanol) evaporates more readily. The vapours formed will move further up the column where they will be condensed again due to the lower temperature.
- This process of evaporation and condensation is repeated many times in the column, the vapours moving up the column become richer in the liquid with lower boiling point (ethanol).
- Eventually, the vapour of the liquid with lower boiling point (ethanol) will be able to reach the top of the column first and be distilled over into the receiver.

the glass beads in

- In short, a fractionating column provides **large surface area for condensation of vapour**.

Applications:

a. Crude oil - separated into different fractions (e.g. petrol, diesel) having different boiling point ranges. Different fractions have different uses.

b. Liquid air - Air is cooled until it is liquefied and then separated into nitrogen, oxygen, argon etc.

c. Fermented liquor - The dilute ethanol solution obtained after fermentation of sugar is concentrated and purified by fractional distillation.

2.7 Use of a separating funnel

Use : To separate two immiscible liquids from each other.

- Immiscible liquids are liquids that do not dissolve in each other.

Method : 1. The mixture is placed in the funnel and allowed to settle.

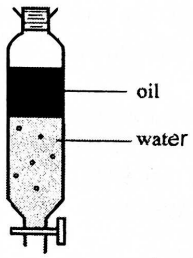
2. The liquids will then separate into two layers - the denser layer being on the bottom.

3. The stopper is removed and the tap is opened. The lower layer liquid is then allowed to flow out first and is collected in a container.

4. The tap is closed just before the interface of the two liquids is reached. The interface portion is discarded.

5. The tap is then opened again to run the upper layer liquid into another container.

Example : To separate oil/petrol from water.



2.8 Chromatography

Use : To separate two or more components that dissolve in the same solvent.

- There are many types of chromatography. They include Paper Chromatography, Thin- layer Chromatography (TLC), Liquid Chromatography (LC) and Gas Chromatography (GC) etc.
- Chromatography may be used to test the purity of a substance if
  - the substance cannot be melted/ boiled easily,
  - the amount of substance is very small.

Paper Chromatography

Method : 1. On a piece of chromatography paper, draw a line using a pencil.

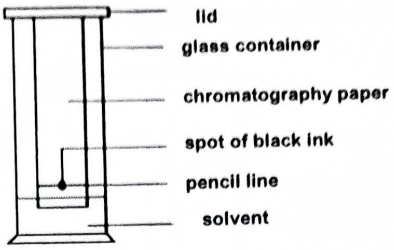
2. Put a small drop of sample on the pencil line.

3. Place the paper into a glass container with a suitable solvent.

4. Allow the apparatus to stand until the solvent nearly reaches the top of the paper.

- The chromatography paper with the separated components is called a chromatogram.
- The solvent front is the position reached by the solvent.
- A **pure** substance always gives a single spot on a chromatogram.
- An **impure** substance gives more than one spot on a chromatogram.

Example : To separate black ink into its component dyes



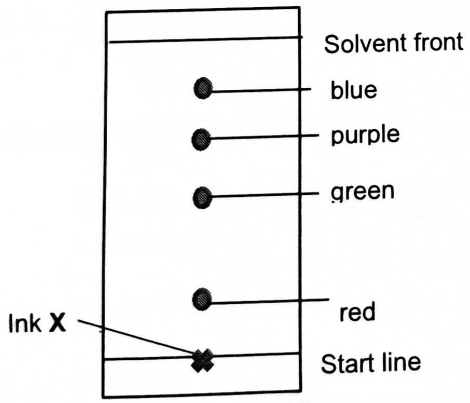
Precautions for Paper Chromatography

- The start line must be drawn in pencil and not in ink as ink may dissolve in the solvent and interfere with the results but pencil lead is insoluble in the solvent.
- The start line must be above the solvent level at the start of the experiment to prevent the samples from dissolving in the solvent.
- The spots on the start line must be small to prevent overlapping and smudging when they travel up the paper.
- The solvent front must be allowed to reach near the top of the chromatography paper to ensure complete separation of mixture into its components.
- A lid is needed for the container to prevent the evaporation of the solvent especially when the solvent is volatile (e.g. ethanol).

Principles of Chromatography

- Different components in the mixture have different solubilities in the solvent.
- The more soluble components will get carried further up the paper by the moving solvent compared to the less soluble ones. Hence, the components in the mixture travel different distances on the paper and are separated.

A black ink is separated into its dyes by paper chromatography. The following chromatogram is obtained.



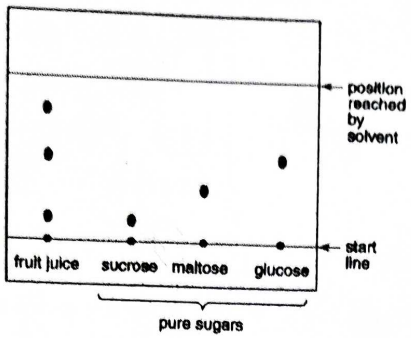
The chromatogram shows that

- Ink **X** is a mixture consisting of 4 dyes.
- Blue dye is the most soluble in the solvent used while red dye is the least soluble.

- Chromatography can be used to identify an unknown substance by
  - comparing its chromatogram with that of pure known substances prepared under the same conditions. Identical substances will travel up the same distance on the chromatography paper.
  - comparing its R_f values with that of pure known substances (chromatograms must be obtained under the same conditions)

Example:

The following is a chromatogram of sugars in a fruit juice:



Q1 How many different sugars does the fruit juice contain?

3

Q2 What are the sugars present in fruit juice?

Sucrose and glucose

Q3 Sugars are colourless substances, how to make them visible on chromatogram?

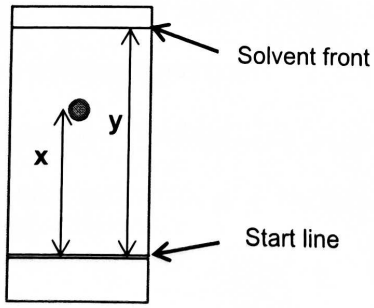
The chromatogram is sprayed with a locating agent. A locating agent is a chemical that reacts with the colourless substances to form coloured products.

R_f value (Retention factor)

- The R_f value for any substance on
- a chromatogram

$$= \frac{\text{distance moved by the substance}}{\text{distance moved by the solvent}} = \frac{x}{y}$$

x : distance measured from the start line to the centre of the spot
y : distance measured from from the start line to the solvent front

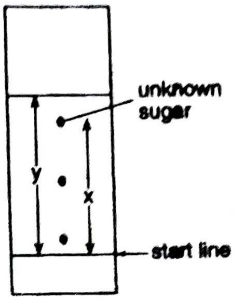


- The R_f value of a substance is a constant as long as chromatography is carried out under the same conditions (i.e. same solvent and same temperature). This value can be used to identify substances.

Quick Check

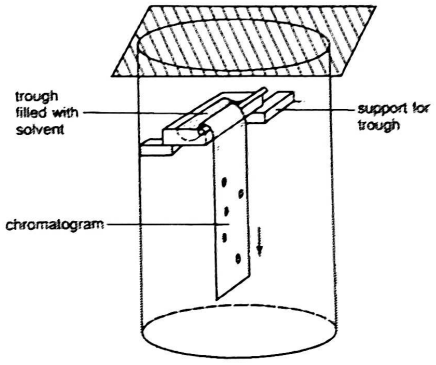
Identify the unknown sugar in the following chromatogram by calculating its R_f value and compare it with the given R_f values of sugars in the table.

Sugar	R_f values
fructose	0.86
glucose	0.57
maltose	0.40
sucrose	0.20
galactose	0.69



[Ans: fructose]

The diagram below shows the descending method of chromatography which is carried out with the solvent running down the paper.



Q State one advantage of using descending method of chromatography.

This method works better for longer piece of paper as the solvent flows more quickly since it does not have to move against gravity. A longer piece of paper ensures a more complete separation of the solutes in the mixture.

Advantage of Chromatography

- It is able to detect very small amount of substances , that is less than 10^{-12} g of the substance.

Applications:

a. Detection and identification of drugs in urine samples (illegal drugs used by athletes for improving their performances or for identifying drug addicts)

b. Detection and identification of banned substances in food (e.g. poisonous artificial dyes)

c. Separation of mixtures of dyes, sugars and amino acids

3 Criteria of Purity

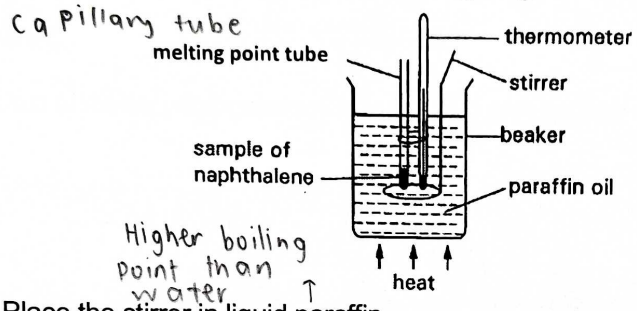
- The following 3 techniques can be used to
  - identify a substance
  - test the purity of a substance

3.1 Finding the Melting Point (For solids)

Example : Finding the melting point of naphthalene

Method :

1. Put the test solid (naphthalene) into the melting point tube and attach it to the bulb of a thermometer as shown in the following diagram:



Higher boiling point than water ↑

2. Place the stirrer in liquid paraffin.
3. Heat gradually with constant stirring to keep the temperature uniform.
4. Record the temperature as the solid sample starts to melt. Record the temperature again when the sample has fully melted.

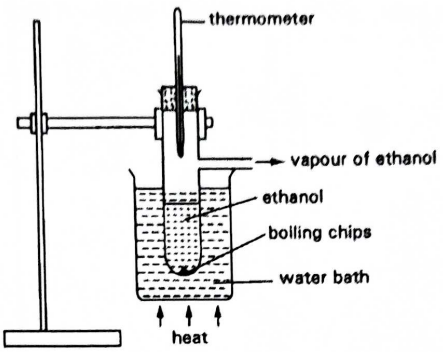
- A **pure** solid has a **fixed** melting point. It melts completely at a constant temperature.
- If the solid is **impure**,
  - its melting point is lowered.
  - it melts over a range of temperatures.
- Pure naphthalene will melt completely at a constant temperature of 80°C.
- Impure naphthalene might start melting at 76°C but the melting may not be complete until the temperature reaches 79°C. Hence, we say that impure naphthalene melts over a range of temperature.

3.2 Finding the Boiling Point (For liquids)

Example : Finding the boiling point of ethanol

Method :

1. Pour approximately 5 cm^3 of test liquid (ethanol) into a boiling tube with a side arm.
2. Set up the apparatus as shown in the following diagram:



test tube

flame

test tube might explode as the pressure may be too high

3. Heat the liquid gradually until the liquid starts to bubble and a constant temperature is shown on the thermometer.

Note:

- Ethanol is flammable and hence a water bath is preferred over direct heating of the liquid
- The boiling tube has a side arm to allow vapour of ethanol to escape to prevent the pressure from building up in the tube.

- A **pure** liquid has a **fixed** boiling point (at one atmosphere pressure). It boils at a constant temperature.
- The thermometer should read a constant temperature of 78°C if ethanol is pure.

3.3 Using Chromatography

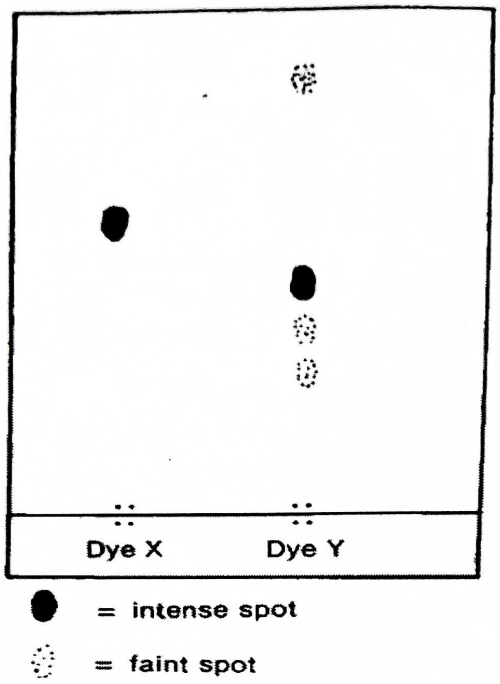
Chromatography may be used to test the purity of a substance if

* the substance cannot be melted/ boiled easily,
* the amount of substance is very small.

- A **pure** substance **always** gives a **single** spot on a chromatogram.
- An **impure** substance gives **more than one spot** on a chromatogram.

Example:

Testing the purity of two dyes (**X** and **Y**) used in food using paper chromatography.



* Dye **X** is pure as it forms only one spot on chromatogram.
* Dye **Y** is impure as it has separated into several spots on the chromatogram.
* The intense spot is the main component in dye **Y**.
* The three faint spots are impurities in dye **Y**.

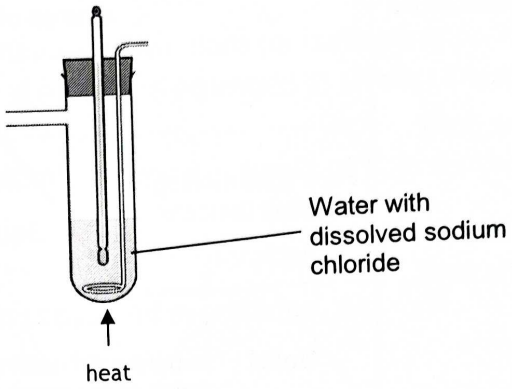
$$\text{Rf value} = \frac{\text{Distance travelled by spot}}{\text{Distance travelled by solvent}}$$

(less than or equal to 1)

Determining boiling point of an impure liquid (containing solid impurity)

- If the liquid is **impure**,
  - its boiling point is raised,
  - it boils over a range of temperatures.

- The apparatus below can be used to find the boiling point of water with dissolved sodium chloride as impurity.



Q1 Which is the likely temperature on the thermometer when the impure water in the above boiling tube begins to boil?

A 96°C **B** 99°C **C** 100°C **D** 102°C

(D) 102°C

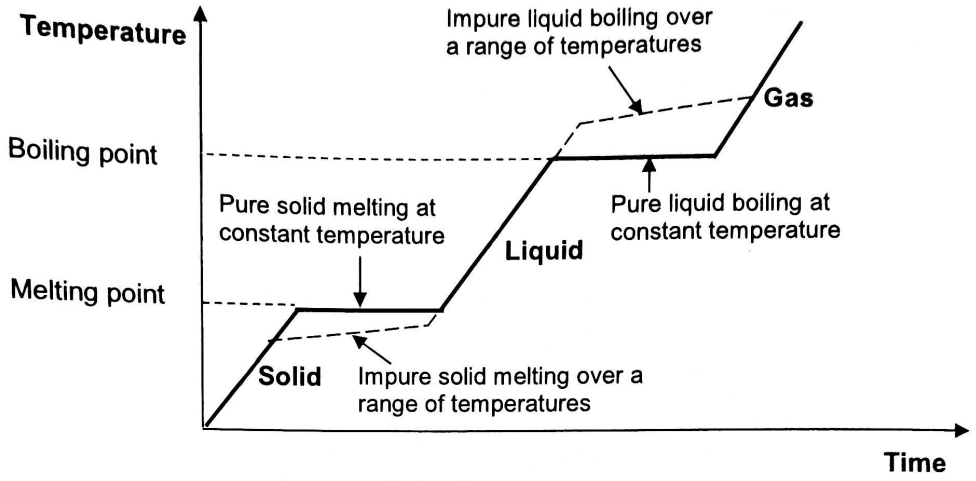
Sodium chloride impurity will increase the boiling point of water to above 100°C.

Q2 What would be the reading on the thermometer if it is placed above the boiling mixture?

100°C. When the thermometer is placed above the mixture, it will record the temperature of steam. During boiling, only the pure water vaporises, rises and escapes from the tube. Sodium chloride salt will remain in the tube.

Heating curve

- By taking the temperature of the substance at regular intervals when a solid is heated until melts and then boils to form the vapour, a heating curve of the substance may be obtained.



- If a substance is pure, the heating curve shows a constant melting point and constant boiling point.
- Impurity lowers the melting point and raises the boiling point and each change of state occurs over a range of temperatures.

Quick Check

An impure sample of substance **X** melts at about 113°C. The table below gives a list of substances with a melting point near 113°C. Which of the substance could be **X**?

Substance	Melting point of pure substance (°C)
Resorcinol	111
Catechol	105
Nitrophenol	114
Butanamide	115

*Impurities lower the melting point of **X**. Hence actual melting point of **X** must be above 113°C, so **X** could be nitrophenol or butanamide.*