1 Experimental Chemistry

Study Station ▸▸

A How Are Physical Quantities Measured and Gases Collected in Experiments?

Learning Outcomes

- Name appropriate apparatus for the measurement of time, temperature, mass and volume.
- Suggest suitable apparatus for simple experiments.
- Suggest suitable apparatus for the collection of gases.

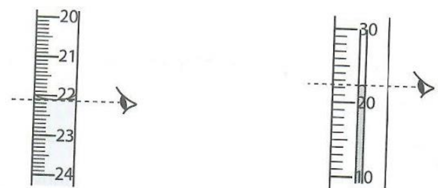
Physical Quantities

1. The **International System of Units (SI Units)** is used as a common standard for measurements.
2. Some **physical quantities** used in Chemistry experiments are shown below.

Physical Quantity	mass	time	temperature	volume
SI Unit	kilogram (kg)	second (s)	kelvin (K)	cubic metre (m^3)
Other Units	• gram (g) • tonne (t)	• minute (min) • hour (h)	• degree Celsius (°C)	• cubic centimetre (cm^3) • cubic decimetre (dm^3)
Conversion of Units	• 1 kg = 1000 g • 1 t = 1000 kg	• 1 min = 60 s • 1 h = 60 min	• T (K) = T (°C) + 273	• 1 m^3 = 1000 dm^3 • 1 dm^3 = 1000 cm^3
Apparatus	• beam balance • electronic balance (accuracy: ±0.01 g)	• analogue stopwatch (accuracy: ±0.1 s) • digital stopwatch (accuracy: ±0.01 s)	• analogue thermometer, e.g. mercury, alcohol • digital thermometer • temperature sensor / probe connected to a data logger	For liquids: • measuring cylinder (measures volumes to the nearest 0.5 cm^3) • burette (measures volumes to the nearest 0.05 cm^3) • pipette (measures fixed volumes, e.g. 10.0 cm^3 or 25.0 cm^3) For gases: • gas syringe (measures volumes up to 100 cm^3)

Reading the Volume of a Liquid

1. When a liquid is placed in a container, it forms a curve called the **meniscus**.
   - Most liquids, such as water, have a **concave** meniscus, which curves upwards at the edges.
   - Mercury has a **convex** meniscus which curves downwards at the edges.
2. The eye should be aligned to the meniscus when reading the volume of a liquid. This prevents **parallax error**.



reading off the bottom of a concave meniscus

reading off the top of a convex meniscus

Worked Example 1.1

What apparatus should be used to measure each of the following?

(a) The time taken for calcium carbonate powder to dissolve in hydrochloric acid

(b) 10.0 cm^3 of sulfuric acid

(c) 16.85 cm^3 of aqueous potassium hydroxide

(d) 65.0 cm^3 of oxygen gas

(e) 2.35 g of salt

Strategy

(a) The apparatus used should be able to measure time accurately.

(b) The apparatus used should be able to measure fixed volumes of liquids such as 10.0 cm^3.

(c) The apparatus used should be able to measure a volume of liquid to the nearest 0.05 cm^3.

(d) The apparatus used should be able to measure the volume of a gas.

(e) The apparatus used should be able to measure mass to the nearest 0.01 g.

Solution

(a) Digital stopwatch

(b) Pipette

(c) Burette

(d) Gas syringe

(e) Electronic balance

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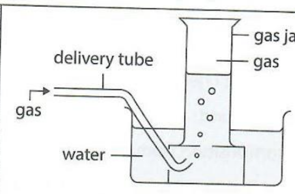
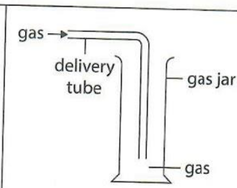
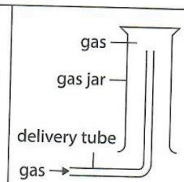
Collecting a Gas

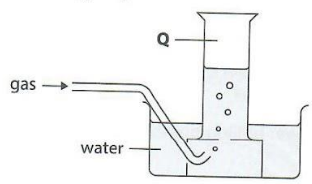
1. The method of collecting a gas depends on how soluble the gas is in water and how dense the gas is compared to air.
2. Some methods of collecting a gas are shown below.

Method of Collecting a Gas	 displacement of water	 downward delivery of gas	 upward delivery of gas
Properties of Gas Collected	• insoluble in water, e.g. hydrogen • slightly soluble in water, e.g. oxygen, carbon dioxide	• soluble in water, e.g. chlorine • very soluble in water, e.g. hydrogen chloride, sulfur dioxide • denser than air	• very soluble • less dense than air, e.g. ammonia

3. An inverted measuring cylinder that is filled with water can also be used to measure the volume of a gas through the displacement of water.
4. A **gas syringe** can be used to collect a gas if the volume of the gas needs to be measured.

Worked Example 1.2

The apparatus below is used to collect gas **Q**.



What could **Q** be?

A Ammonia

B Carbon dioxide

C Hydrogen chloride

D Sulfur dioxide

Solution

B

Explanation

Q must be insoluble in water so that it can be collected by displacement of water. Carbon dioxide does not dissolve in water.

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B How Are Substances in Mixtures Separated?

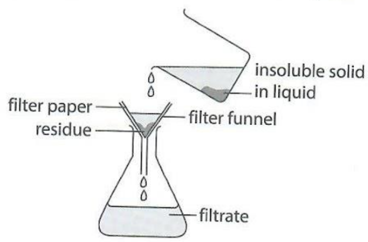
Learning Outcomes

- Describe how the components of mixtures can be separated and purified.
- Suggest how different types of mixtures can be separated and purified.
- Interpret paper chromatograms.

1. A **mixture** is made up of two or more substances that are not chemically combined.
2. A **pure** substance is made up of only one element or compound.
3. A substance with **impurities** has a different chemical composition from the pure substance.
4. There are several methods to remove impurities from a substance or separate the constituents of a mixture. The properties of each substance in the mixture determine the method to be used.

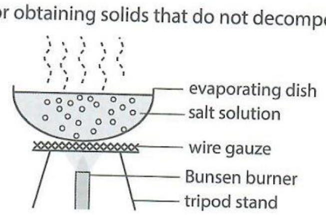
Separating Solid–Liquid Mixtures

1. **Filtration** can be used to separate an insoluble solid from a liquid.



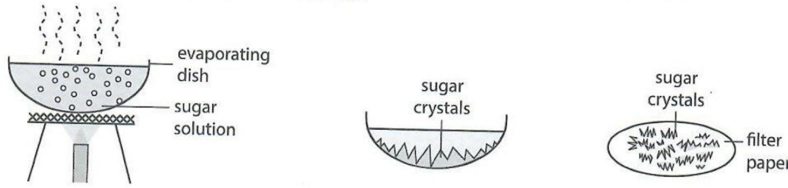
- The insoluble solid is collected as the **residue**.
- The liquid is collected as the **filtrate**.

2. **Evaporation to dryness** can be used to obtain a soluble solid from a solution by heating the solution until all the solvent boils off.
   - The solvent which has a lower boiling point than the solute changes into a gas first. The solute is left behind.
   - This method is suitable for obtaining solids that do not decompose on heating, e.g. salt.



3. **Crystallisation** can be used to obtain a soluble solid from a solution by allowing a hot saturated solution to cool.
   - A **saturated solution** is a solution in which no more solute can be dissolved in the solvent.
   - This method is suitable for obtaining solids that decompose on heating, e.g. sugar.
   - Many crystals require a fixed amount of water in its crystal lattice. This water is known as water of crystallisation. If the water is driven off by evaporation to dryness, a powder instead of crystals will be obtained. Thus, crystallisation is used to obtain crystals.

4. The steps of crystallisation are shown below.

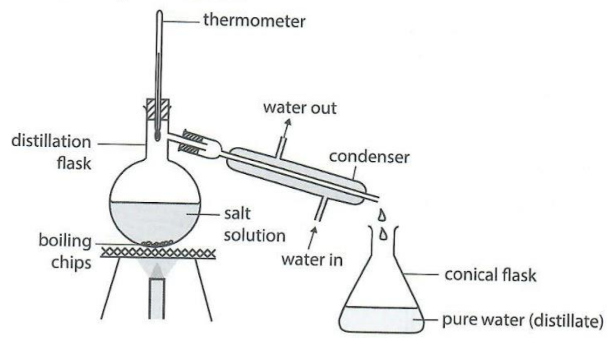


(a) Heat the solution until it is saturated.

(b) Leave the hot saturated solution to cool until crystallisation occurs.

(c) Filter to collect the crystals. Wash the crystals with cold distilled water and dry them between a few sheets of filter paper.

5. **Simple distillation** can be used to separate a solvent from a solute. For example, water can be separated from salt using this method.



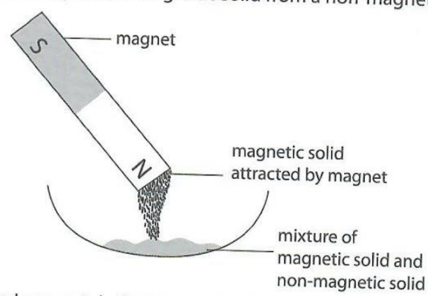
- The solvent which has a lower boiling point than the solute changes into a vapour first. The solute remains in the distillation flask.
- The vapour reaches the top of the distillation flask and enters the condenser.
- The vapour cools in the condenser and condenses back into a liquid.
- The liquid is collected in the conical flask as the **distillate**.
- Once all the solvent has changed into a vapour, the solute is left in the distillation flask as the residue.

Tip

Distillation can also be used to separate a mixture of liquids.

Separating Solid–Solid Mixtures

1. A **magnet** can be used to separate a magnetic solid from a non-magnetic solid.

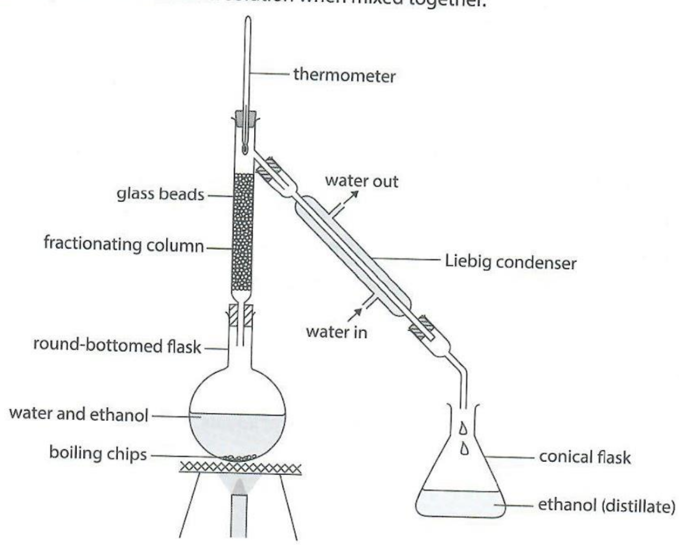


- Iron, cobalt and nickel are metals that are magnetic.
- Compounds of some metals are also magnetic. For example, magnetite, a naturally occurring oxide of iron is magnetic.

2. A soluble solid can be separated from an insoluble solid by dissolving the soluble solid in a suitable solvent and carrying out filtration, followed by evaporation to dryness or crystallisation.
   - The insoluble solid is collected as the residue.
   - The soluble solid is obtained from the filtrate by evaporation to dryness or crystallisation.

Separating Liquid–Liquid Mixtures

1. **Fractional distillation** can be used to separate miscible liquids with different boiling points. **Miscible** liquids form a uniform solution when mixed together.



- When a mixture containing two miscible liquids is heated, both liquids change into vapours.
- The vapour of the liquid with the higher boiling point condenses into a liquid in the fractionating column and flows back into the round-bottomed flask.
- The vapour of the liquid with the lower boiling point reaches the top of the fractionating column and enters the condenser. It condenses into a liquid in the condenser.
- The liquid with the lower boiling point is collected first in the conical flask as the distillate.
- When all the liquid with the lower boiling point has distilled over, the temperature in the fractionating column increases to reach the boiling point of the remaining liquid.
- The liquid with the higher boiling point distils over and can be collected separately.

2. During fractional distillation, the thermometer measures the temperature of the vapour that enters the condenser. It does not measure the temperature of the liquid in the round-bottomed flask, which may be affected by the heat source.

Common Misconception

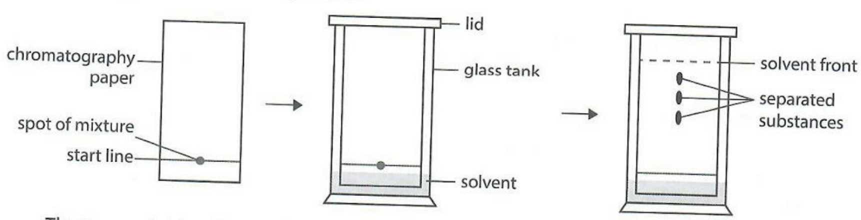
✗ Unlike fractional distillation, simple distillation cannot be used to separate a mixture of miscible liquids.

✓ Like fractional distillation, simple distillation can be used to separate a mixture of miscible liquids.

Explanation

Distillation can be used to separate miscible liquids as long as there is a significant difference in the boiling points of the liquids, i.e. more than 50 °C.

3. **Chromatography** can be used to separate a mixture of substances which have different solubilities in a given solvent. The substances to be separated are distributed between two phases, one of which is stationary and does not move, while the other phase moves in a definite direction.
4. In **paper chromatography**, the solvent moves up the stationary chromatography paper, carrying with it the substances to be separated.

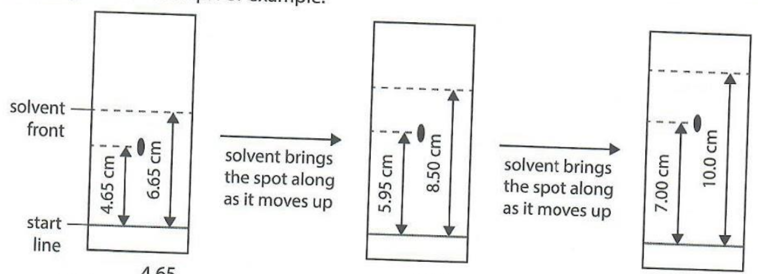


- The more soluble substances move more quickly with the solvent than the less soluble substances. As a result, the substances in the mixture are separated.
- The chromatography paper with the separated substances is called a **chromatogram**.

5. The ratio between the distance travelled by a substance and the distance travelled by the solvent is a constant known as the **retention factor**, or $\boldsymbol{R_f}$ **value** of the substance.

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

6. The distances travelled by the substances and the solvent increase with the time given for the chromatogram to develop. For example:

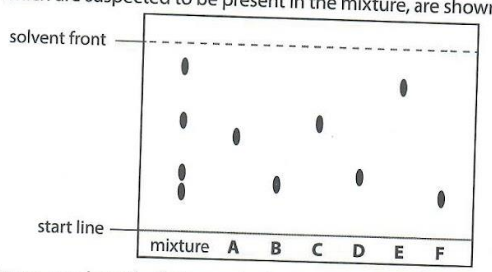


R_f value $= \frac{4.65}{6.65} = 0.70$ R_f value $= \frac{5.95}{8.50} = 0.70$ R_f value $= \frac{7.00}{10.0} = 0.70$

7. The R_f value of a substance does not change as long as the chromatogram is obtained under the same conditions, i.e. same solvent and same temperature. This allows substances to be identified.

Worked Example 1.3

A mixture is analysed using paper chromatography. The chromatogram of the mixture and six pure compounds, **A** to **E**, which are suspected to be present in the mixture, are shown below.



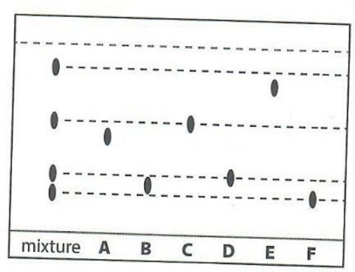
(a) What is the minimum number of substances in the mixture?

(b) Which compounds are identified to be present in the mixture?

(c) One of the substances in the mixture **cannot** be identified. Circle this substance in the diagram.

Strategy

Draw a line from the centre of the spot for each substance, parallel to the start line and solvent front. The centre of the spot for a pure compound will coincide with one of the lines if the pure compound is found in the mixture. The distance moved by a spot is also taken from the centre of the spot to the start line.

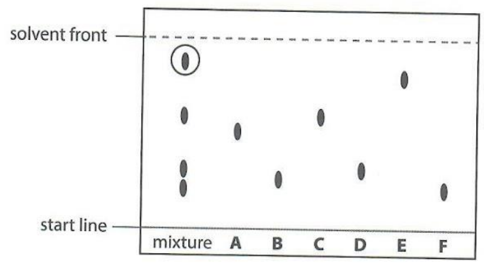


Solution

(a) Four

(b) **C**, **D** and **F**

(c)



Worked Example 1.4

A sample of sugar is contaminated with sand. Describe the steps you would take to purify the sugar.

Strategy

Sugar is soluble in water while sand is insoluble. The sugar can be dissolved in water and the sand can be removed by filtering the solution.

Solution

Dissolve the sample of sugar in the minimum amount of water. Ensure that the maximum amount of sugar dissolves in the water. Filter the mixture and collect the filtrate. Heat the filtrate until a hot saturated solution is obtained. Allow the hot saturated solution to cool slowly and form sugar crystals. Filter the solution. Wash the sugar crystals with distilled water and dry them between sheets of filter paper.

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C How Can the Purity of Substances Be Determined?

Learning Outcome

- Deduce the identities of substances and their purity based on their melting and boiling points.

1. Measuring the **purity** of substances is important as it helps to identify harmful impurities and ensures that products such as foodstuffs and drugs meet quality standards.
2. Purity can be determined based on the following:
   (a) Use of chromatography
   - A pure substance will only give a single spot in the chromatogram.
   - Impurities will appear as multiple spots with different R_f values on the chromatogram.

   (b) Melting and boiling points of a substance
   - A pure substance has constant and fixed melting and boiling points.
   - Impurities lower the melting point of the substance and raise its boiling point.
   - The more impurities present, the lower the melting point of the substance and the higher its boiling point.

Worked Example 1.5

Which of the following are possible methods to determine whether a substance is pure?

1 Determining whether the boiling point of the substance is constant
2 Determining whether the freezing point of the substance is constant
3 Obtaining a chromatogram of the substance using a suitable solvent

A 1 only
B 1 and 2 only
C 1, 2 and 3
D 2 and 3 only

Solution

C

Explanation
A pure substance has constant and fixed boiling and melting points. The freezing point of a substance in the liquid state is the same as its melting point when it is in the solid state. A pure substance will give only a single spot in the chromatogram.

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Checkpoint 1.1

1. Study Figure 1.1.

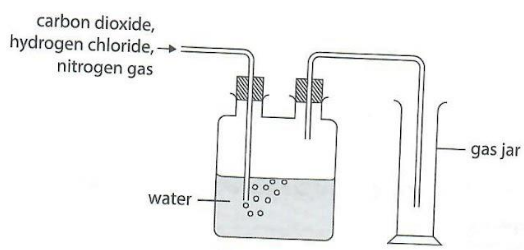

Figure 1.1

(a) Which gas will be collected in the gas jar?
(b) Explain your answer in **(a)**.

2. Describe how the components of the following mixtures can be separated.
(a) Ethanol and water
(b) Pigments in a whiteboard marker
(c) A solution made up of sugar and water

3. Students **A** and **B** were given samples of a compound and asked to determine the purity of the compound using chromatography. They obtained the chromatograms at the same room temperature as shown in Figure 1.2.

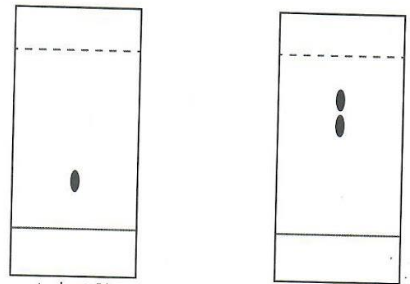

Figure 1.2

(a) Suggest why the two chromatograms looked different.

(b) Student **A** claimed that the compound is pure.

(i) Suggest why she made this claim.

(ii) Suggest **one** reason why her claim may not be true.

(c) Student **B** concluded that there is at least one impurity in the sample. Suggest how he can determine which spot on his chromatogram belongs to the impurity.

Test Station ▸▸

1. Which of the following apparatus is **most** suitable for measuring 5.85 cm^3 of a solution?

A 10 cm^3 measuring cylinder

B 10 cm^3 pipette

C 20 cm^3 beaker

D 50 cm^3 burette

2. The melting and boiling points of pure citric acid are 156 °C and 310 °C respectively.

What are likely to be the approximate melting and boiling points of an **impure** sample of citric acid?

	Melting Point / °C	Boiling Point / °C
A	153	307
B	154	312
C	158	308
D	160	313

3. An unknown compound, **K**, was found to be one of four substances, **L**, **M**, **N** and **P**. Two chromatograms of **K**, **L**, **M**, **N** and **P** were obtained using two different solvents, **X** and **Y**.

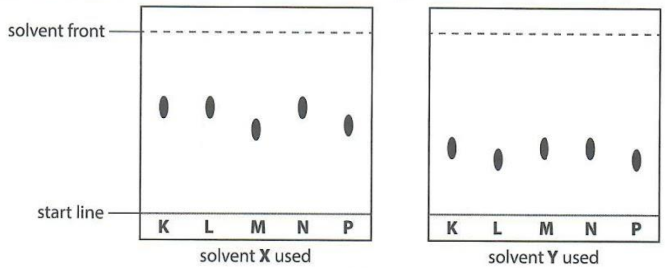

Figure 1.3

What is the identity of **K**?

A **L**

B **M**

C **N**

D **P**

4. Study the information in Table 1.1.

Table 1.1

Substance	Boiling Point / °C	Behaviour in Water
water	100	–
silver chloride	1547	insoluble
sodium chloride (salt)	1465	soluble
isobutanol	108	miscible
ethanal	20	miscible

Based on the information in Table 1.1, suggest how the following substances can be separated from water.

(a) Silver chloride [2]

(b) Sodium chloride [2]

(c) Isobutanol [2]

(d) Ethanal [2]

5. Two-dimensional paper chromatography can be used to separate complex mixtures. In this method, paper chromatography is carried out first. After that, the chromatogram is turned 90° anticlockwise and paper chromatography is carried out a second time using a different solvent.

Figure 1.4 shows the chromatogram obtained when a mixture is subjected to two-dimensional paper chromatography.

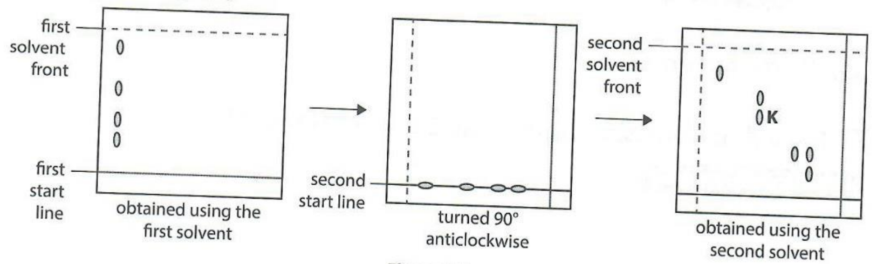

Figure 1.4

(a) Suggest why the chromatogram is run a second time using a different solvent. [1]

(b) (i) State the number of components in the mixture. [1]

(ii) The R_f value of substance **K** when the first solvent is used is slightly greater than the R_f value when the second solvent is used.

Label, in Figure 1.4, the distance travelled by substance **K** as "x" and the distance travelled by the solvent as "y" when each solvent is used. [2]

(iii) Sketch the chromatogram that is obtained using the second solvent only. [1]

(c) The second chromatogram can be turned 90° anticlockwise and a third chromatogram obtained using another solvent.

(i) What is **one** difference in calculating the R_f values of the components for this third run? [1]

(ii) Based on your answer in **(c)(i)**, what is **one** disadvantage of running the chromatogram a third time? [1]