Cambridge OL

Chemistry

CODE: (5070)

Chapter 14

Experimental techniques and chemical analysis

14.1 Apparatus used for measurement in chemistry

Scientists study materials through laboratory experiments using scientific apparatus for measurements. Understanding proper use is crucial for successful experimentation and investigation planning. Common measurements in chemistry labs include temperature, mass, volume of liquids and gases, and time. Each method has advantages and disadvantages, so selecting the appropriate apparatus requires knowledge.

Choosing the appropriate method or apparatus requires understanding its advantages and disadvantages to determine its suitability. You will have to decide:

» What apparatus should you use to measure each of these?

» Which piece of apparatus is most suitable for the task?

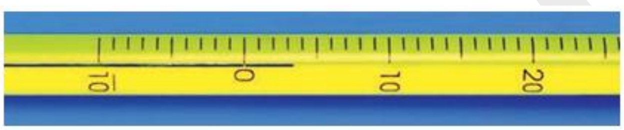
» How do you use the piece of apparatus correctly? Before you use a piece of apparatus, be sure you know how to use it properly. You need to be safe in your working habits and also to ensure you use good techniques,

Measurement of time

Experiments involving rates of reaction will require the use of a stopwatch (Figure 14.1) that measures to a hundredth of a second. The units of time are hours (h), minutes (min) and seconds (s).

▲ **Figure 14.1** This stopwatch can be used to measure the time passed in a chemical reaction. The reading is 6.3 s

Measurement of temperature



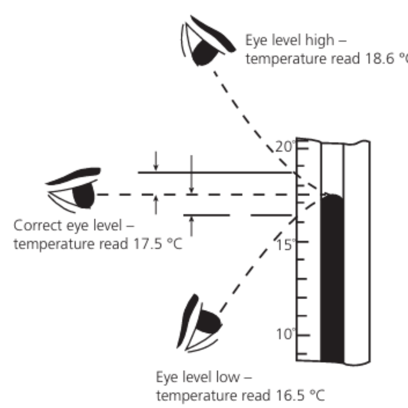
▲ **Figure 14.2** A thermometer can be used to measure temperature. The reading is 3°C

This scale is based on the temperature at which water freezes and boils, that is:

» The freezing point of water is 0°C

» The boiling point of water is 100°C.

The thermometer, as shown in Figure 14.2, accurately measures between -10°C and 110°C at 1°C intervals. It can be read to the nearest 0.5°C if the reading is between two scale marks. Ensure eye level is at same level.



▲ **Figure 14.3** Eye should be level with the liquid meniscus

Measurement of mass

Electronic balances measure mass in grams and kilograms, with precision based on the smallest mass measured on the scale setting. Wait until steady reading before taking it.

Measurement of volume of liquids

Different liquid experiments require different measuring apparatuses for volume measurement. Figure 14.5 displays three commonly used measuring apparatuses for liquid volume.

Acid-base titration

A burette and a **pipette** are needed in a titration. This technique is often used to test the strength or purity of an acid or an alkali.

A titration involves adding a known concentration of a solution, such as an acid, to an unknown concentration of another solution, such as an alkali, in a flask. The **titrant** in the burette indicates the endpoint, while the pipette is used to measure the known volume in the flask.

A measuring cylinder, also known as a mixing or graduated cylinder, is a laboratory tool used to measure the volume of a liquid, measured in units like litres, cubic decimetres, or cubic centimetres.

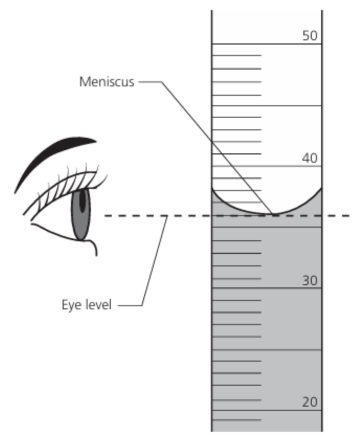
$$1 \text{ litre} = 1 \text{ dm}^3 = 1000 \text{ cm}^3$$

Some manufacturers use millilitres (ml) for volume measurements. To read volume, ensure the apparatus is vertical and your eye is level with the liquid's meniscus. The precision of the measurement varies depending on the apparatus, with burettes having a 0.1 cm^3 precision and the bottom of the meniscus between divisions.

Measurement of volume of gases

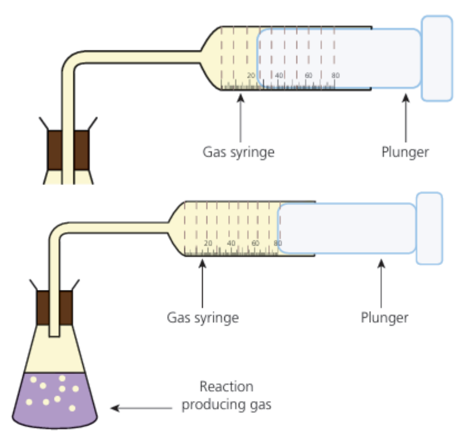
The volume of a gas can be measured with a gas syringe. This is used to measure the amount of gas collected in experiments. They have a maximum volume of 100 cm^3 (Figure 14.7).

▲ **Figure 14.7** A gas syringe



▲ **Figure 14.6** The volume level in this measuring cylinder should be read on the dotted line. The reading here is 36 cm^3

In certain reactions, the reaction rate can be monitored by collecting the volume of gas generated over time. A gas syringe connected to the gas flask has a scale that allows measuring the collected volume. A graph plotting this volume against time shows the reaction rate.



▲ **Figure 14.8** As gas is collected during the reaction, the plunger is forced out and the volume can be read from the scale on the side

14.2 Separating mixtures

Chemists separate useful substances from impurities in mixtures, using various methods based on the mixture's properties, substances' properties, and whether they are solids, liquids, or gases.

Separating solid/liquid mixtures

If a solid substance is added to a liquid it may dissolve to form a **solution**. In this case the solid is said to be soluble and is called the **solute**. The liquid it has dissolved in is called the **solvent**.

Key definitions

A **solvent** is a substance that dissolves a solute.

A **solute** is a substance that is dissolved in a solvent.

A **solution** is a liquid mixture composed of two or more substances.

Filtration

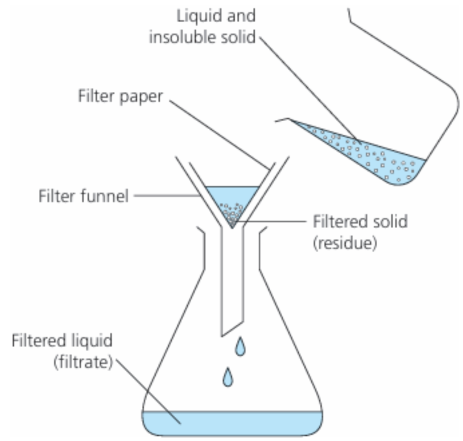
Filtration is a common separation technique in chemistry laboratories, used to separate solids from liquids, such as sand from water, by pouring a cup of tea through a strainer.

The filter paper acts as a sieve, trapping sand particles in small holes. Water molecules pass through, leaving the **residue**, which is the water, and the **filtrate**, which is the water.

Key definitions

Residue is a substance that remains after evaporation, distillation, filtration or any similar process.

Filtrate is a liquid or solution that has passed through a filter.



▲ **Figure 14.9** It is important when filtering not to overfill the filter paper

Evaporation

If a solid has dissolved in a liquid it cannot be separated by filtering or centrifuging. Instead, the solution can be heated so that the liquid evaporates completely and leaves the solid behind.

Crystallization

Salt is obtained from sea water using Sun's heat to evaporate water, leaving a **saturated solution** called brine. This solution contains the maximum concentration of a solute, with solubility varying with temperature. As the solution becomes saturated, salt **crystallizes** and is removed using large scoops.

Key definition

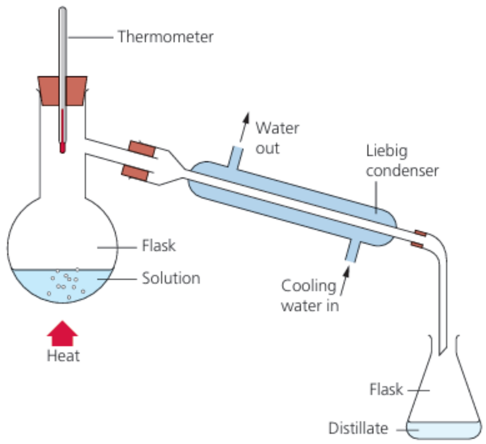
A **saturated solution** is a solution containing the maximum concentration of a solute dissolved in the solvent at a specified temperature.

▲ **Figure 14.12** Apparatus used to slowly evaporate a solvent

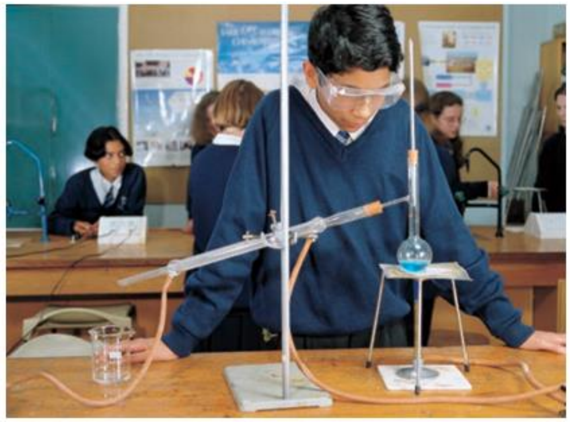
▲ **Figure 14.13** Salt is obtained in north-eastern Brazil by evaporation of sea water

Simple distillation

If we want to obtain the solvent from a solution, then the process of distillation can be carried out. The apparatus used in this process is shown in Figure 14.14.



▲ **Figure 14.14** Water can be obtained from salt water by distillation



Separating liquid/liquid mixtures

Oil and water do not mix easily. They are said to be **immiscible**. When cleaning up disasters of this type, a range of chemicals can be added to the oil to make it more soluble. This results in the oil and water mixing with each other. They are now said to be **miscible**.

▲ **Figure 14.16** Millions of litres of oil can be spilled from oil tankers and cleaning up is a slow and costly process

Immiscible liquids

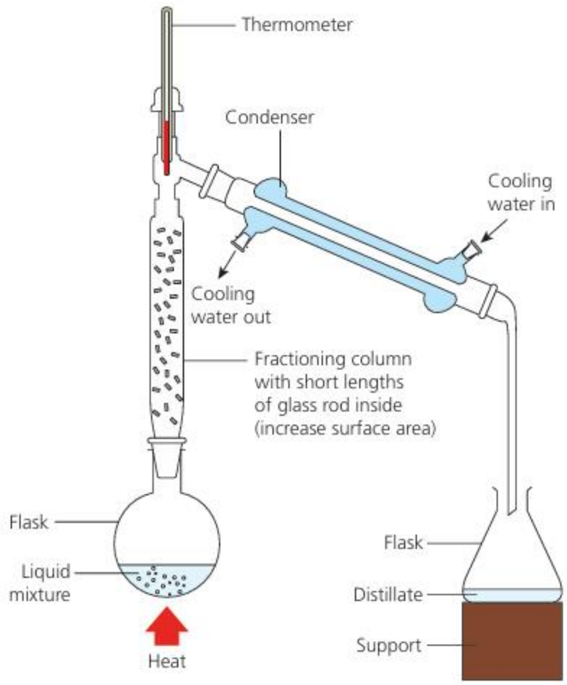
If two liquids are immiscible, they can be separated using a separating funnel. The mixture is poured into the funnel and the layers allowed to separate. The lower layer can then be run off by opening the tap as shown in Figure 14.17

▲ **Figure 14.17** The blue liquid is more dense than the red liquid and so sinks to the bottom of the separating funnel. When the tap is opened the blue liquid can be run off.

Miscible liquids

Fractional distillation is a process used to separate miscible liquids like ethanol and water. It involves heating a mixture, producing vapors with different boiling points. The ethanol boils at 78°C, while water boils at 100°C. The higher boiling point of water causes it to condense with ethanol, resulting in the fractionating column.

The water condenses and drips back into the flask while the ethanol vapour moves up the column and into the condenser, where it condenses into liquid ethanol and is collected in the receiving flask as the **distillate**.



▲ **Figure 14.18** Typical fractional distillation apparatus

▲ **Figure 14.19** Gases from the air are extracted in this fractional distillation plant

Solvent extraction

Sugar and green substances can be extracted from sugar cane and ground-up grass using solvent extraction, were water dissolves sugar and ethanol dissolves green substances.

▲ **Figure 14.22** Cutting sugar cane, from which sugar can be extracted, by using a suitable solvent

Chromatography

Chromatography is a technique used to separate soluble solids, such as inks and dyes, for identification. There are various types, but all follow basic principles. The simplest method is paper chromatography, which involves drawing a baseline on chromatography paper, placing ink on the baseline, and placing the paper in a solvent-containing container with a lid to prevent solvent evaporation.

Chromatography is a method used in medical research and forensic science laboratories to separate various mixtures by analyzing the solubilities of dyes in the solvent and their absorption by the chromatography paper.

Numerical measurements (retardation factors) known as **R_f values** can be obtained from chromatograms. An R_f value is defined as the ratio of the distance travelled by the solute (for example P, Q or R) to the distance travelled by the solvent from the pencil line.

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

Colourless substances can be made visible by spraying the chromatogram with a **locating agent.**

Criteria for purity

Pure substances in chemistry contain a single element or compound not mixed with any other substance. Purity measures the purity of a substance. In the food industry, labeled 'pure' apple juice may contain mixed substances, but purity is essential in the pharmaceutical industry. Drugs are manufactured to high purity, dissolved in a solvent, and subjected to fractional crystallization. Purity affects chemical properties, forming predictable products in chemical reactions. Chemists often use high-purity substances for chemical research in the pharmaceutical, food, and chemical industries.

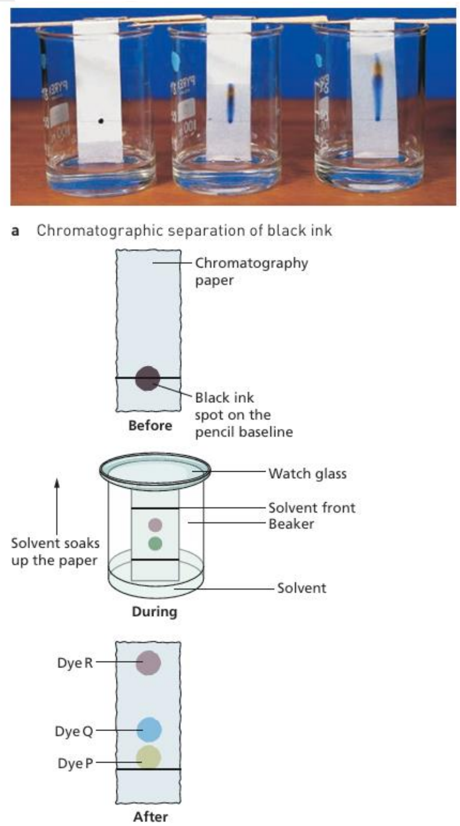
The purity of a substance can be assessed by:

» The melting point of a substance indicates its purity, while if an impurity is present, it indicates a mixture of two or more substances.

» The boiling point of a pure liquid maintains a steady temperature, while an impure substance boils over a temperature range.

»Chromatography produces one well-defined spot for pure substances, while impurities produce several spots on a chromatogram, as shown in Figure 14.23.

▲ **Figure 14.26** These pharmaceuticals must have been through a lot of testing for purity before they can be sold in a pharmacy



b The black ink separates into three dyes: P, Q and R

▲ **Figure 14.23**

14.3 Qualitative analysis

Qualitative chemical analysis is a branch of chemistry that identifies elements or groups in a sample without dealing with quantities. Techniques vary depending on the sample's nature and can be simple or complex. The first stages often require no apparatus and can be observed using color and smell.

Appearance or smell

A preliminary examination of the substance will give you a start. The appearance or smell of a substance can often indicate what it might contain (Table 14.1).

▼ **Table 14.1** Deductions that can be made from a substance's appearance or smell

Observation on substance	Indication
Black powder	Carbon, or contains O^{2-} ions (as in CuO), or S^{2-} ions (as in CuS)
Pale green crystals	Contains Fe^{2+} ions (as in iron(II) salts)
Dark green crystals	Contains Ni^{2+} ions (as in nickel(II) salts)
Blue or blue-green crystals	Contains Cu^{2+} ions (as in copper(II) salts)
Yellow-brown crystals	Contains Fe^{3+} ions (as in iron(III) salts)
Smell of ammonia	Contains NH_4^+ ions (as in ammonium salts)

Flame colours

If a wooden splint, which has been soaked in an aqueous metal ion solution, is held in a colourless Bunsen flame, the flame colour can become coloured (Figure 14.27). Certain metal ions may be detected in their compounds by observing their flame colours (Table 14.2)

▲ **Figure 14.27** The green colour is characteristic of copper

▼ **Table 14.2** Characteristic flame colours of some metal ions

	Metal	Flame colour
Group I (1+ ion)	Lithium	Red
	Sodium	Yellow
	Potassium	Lilac
Group II (2+ ion)	Calcium	Orange-red
	Barium	Light green
Others	Copper (as Cu^{2+})	Blue-green

Flame colours are created by excited electrons in ions absorbing energy from the flame, which is then emitted as visible light, influenced by the electronic configurations of the ions.

Tests for aqueous cations

Effect of adding dilute sodium hydroxide solution

Aqueous sodium hydroxide can identify metal salts like Al^{3+}, Ca^{2+}, Cr^{3+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, and Zn^{2+} by analyzing their color and behavior in excess sodium hydroxide solution.

▼ **Table 14.3** Effect of adding sodium hydroxide solution to solutions containing various metal ions

Added dropwise	To excess	Likely cation
White precipitate	Precipitate is soluble in excess, giving a colourless solution	Al^{3+}, Zn^{2+}
White precipitate	Precipitate is insoluble in excess	Ca^{2+}
Green precipitate	Precipitate is insoluble in excess	Cr^{3+}
Light blue precipitate	Precipitate is insoluble in excess	Cu^{2+}
Green precipitate	Precipitate is insoluble in excess, turns brown near the surface on standing (Figure 14.28)	Fe^{2+}
Red-brown precipitate	Precipitate is insoluble in excess (Figure 14.28)	Fe^{3+}

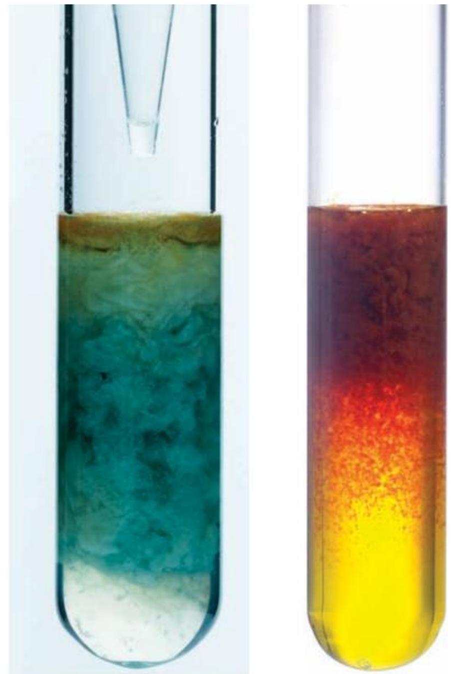
In the case of ammonium salts containing the ammonium ion, NH_4^+, ammonia gas is produced on warming. The ammonium cation does not form an insoluble hydroxide. However, it forms ammonia and water upon heating.

Effect of adding dilute ammonia solution

Aqueous ammonia, a weakly alkaline solution, can identify salts of Al^{3+}, Ca^{2+}, Cr^{3+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, and Zn^{2+} ions by analyzing the precipitate's color.

▼ **Table 14.4** Effect of adding aqueous ammonia to solutions containing various metal ions

Added dropwise	To excess	Cation present
White precipitate	Precipitate is insoluble in excess	Al^{3+}
No precipitate or very slight white precipitate	No change	Ca^{2+}
Green precipitate	Precipitate is insoluble in excess	Cr^{3+}
Light blue precipitate	Precipitate is soluble in excess, giving a dark blue solution (Figure 14.29)	Cu^{2+}
Green precipitate	Precipitate is insoluble in excess, turns brown near the surface on standing	Fe^{2+}
Red-brown precipitate	Precipitate is insoluble in excess	Fe^{3+}
White precipitate	Precipitate is soluble in excess, giving a colourless solution	Zn^{2+}



▲ **Figure 14.28** Addition of sodium hydroxide will show the difference between Fe^{2+} and Fe^{3+} in aqueous solution

Tests for aqueous anions

▼ **Table 14.5** A variety of tests for aqueous anions

Anion	Test	Test result
Carbonate (CO_3^{2-})	Add dilute acid	Effervescence is seen as carbon dioxide produced
Chloride (Cl^-) (in solution)	Acidify with dilute nitric acid, then add aqueous silver nitrate	A white precipitate is produced
Bromide (Br^-) (in solution)	Acidify with dilute nitric acid, then add aqueous silver nitrate	A cream precipitate is produced
Iodide (I^-) (in solution)	Acidify with dilute nitric acid, then add aqueous silver nitrate	A yellow precipitate is produced
Nitrate (NO_3^-) (in solution)	Add aqueous sodium hydroxide, then aluminium foil; warm carefully	Ammonia gas is produced
Sulfate (SO_4^{2-}) (in solution)	Acidify, then add aqueous barium nitrate or barium chloride	A white precipitate is produced (Figure 14.30)
Sulfite (SO_3^{2-})	Add a small volume of acidified aqueous potassium manganite(VIII)	The acidified aqueous potassium manganate(VII) changes colour from purple to colourless

◣ **Figure 14.29** When aqueous ammonia is added to a solution containing Cu^{2+} ions the solution forms a gelatinous light blue precipitate. As more is added to excess, the precipitate dissolves, forming a dark blue clear solution

▲ **Figure 14.30** The test for sulfate ions

Tests for gases

Table 14.6 shows the common gases which may be produced during qualitative analysis and tests which can be used to identify them. These tests are used in conjunction with the tests shown above.

▲ **Figure 14.31** Testing for oxygen gas

▼ **Table 14.6** Tests for gases

Gas	Colour (odour)	Effect of moist indicator paper	Test
Hydrogen (H_2)	Colourless (odourless)	No effect – neutral	'Pops' in the presence of a lighted splint
Oxygen (O_2)	Colourless (odourless)	No effect – neutral	Relights a glowing splint (Figure 14.31)
Carbon dioxide (CO_2)	Colourless (odourless)	Pink – weakly acidic	Turns limewater a cloudy white
Ammonia (NH_3)	Colourless (very pungent smell)	Blue – alkaline	Turns damp red litmus blue
Sulfur dioxide (SO_2)	Colourless (very choking smell)	Red – acidic	Turns acidified potassium dichromate(VI) from orange to green Turns acidified potassium manganate(VII) from purple to colourless
Chlorine (Cl_2)	Yellow-green (very choking smell)	Bleaches moist indicator paper after it initially turns pale pink	Bleaches damp litmus paper
Water (H_2O)	Colourless (odourless)	No effect – neutral	Turns blue cobalt chloride paper pink Turns anhydrous copper(II) sulfate from white to blue

Revision questions

1) a)

The names of some pieces of equipment are shown.

stopwatch balance thermometer gas syringe burette pipette

For each question, name the piece of apparatus that would be used in each case.

Each piece of equipment can be used once, more than once or not at all.

Name the piece of apparatus that would be used to measure the time taken for a white precipitate to form when barium chloride is added to sodium sulfate.

b) Name the piece of apparatus that would be used to determine whether the reaction between magnesium and hydrochloric acid is exothermic.

c) Name the piece of apparatus that would be used to measure the volume of carbon dioxide produced when marble chips are added to hydrochloric acid.

d) Name the piece of apparatus used to add hydrochloric acid to 25 cm^3 of sodium hydroxide solution during a titration.

2) A student investigated the effect of temperature on the rate of reaction between sodium thiosulfate solution and dilute hydrochloric acid:

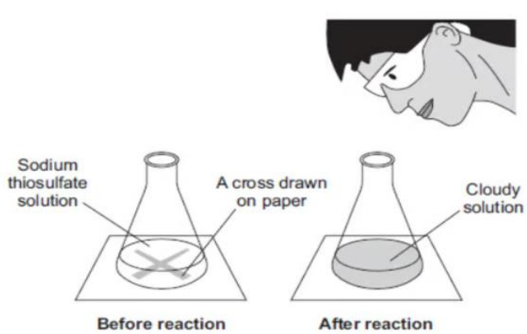
$$Na_2S_2O_3\,(aq) + 2HCl\,(aq) \rightarrow 2NaCl\,(aq) + S\,(s) + SO_2\,(g) + H_2O\,(l)$$

The reaction between these two substances produces a precipitate which makes the mixture turn cloudy.

A student timed how long it took until the cross could no longer be seen through the precipitate, shown in **Figure 1.**

They then calculated the rate of reaction and repeated the experiment at different temperatures.

Figure 1



Name the product that made the mixture go cloudy.

b) Suggest **two** control variables for the investigation that would ensure valid results are obtained.

c) Define the terms:

i) Solution

ii) Solvent

iii) Solute

d) From the products formed in part a) identify:

i) The solute

ii) The solvent

3) Rock salt is a mixture of sand and salt. Salt dissolves in water while sand does not.

A group of students separated rock salt.

They used the following method.

1. Add the rock salt to a beaker.
2. Add 250 cm^3 of water.
3. Leave without disturbing to allow the sand to settle to the bottom.
4. After a while, filter the salt water into an evaporating dish.
5. Heat the evaporating dish with a Bunsen burner until salt crystals begin to form.

i) Suggest **one** improvement to step 2 that the students could have done to make sure all the salt is dissolved.

[1]

ii) What name is given to the solution when no more salt can dissolve at this specific temperature?

b) Identify the residue and filtrate in **step 4**.

c) Suggest **one** safety precaution the students should take in step 5.

4) a) A student investigated the effect of the size of calcium carbonate lumps, $CaCO_3$, on the rate of reaction with hydrochloric acid, HCl, using the following method:

1. Put 50 cm^3 of hydrochloric acid into a conical flask
2. Add 12 g of large calcium carbonate lumps into the flask
3. Attach the gas syringe
4. Measure the volume of gas produced every 30 seconds for 180 seconds
5. Repeat steps 1 - 4 using 12 g of small calcium carbonate lumps.
6. The number of moles of gas for each volume was calculated.

The results for large calcium carbonate lumps are shown below.

Table 1

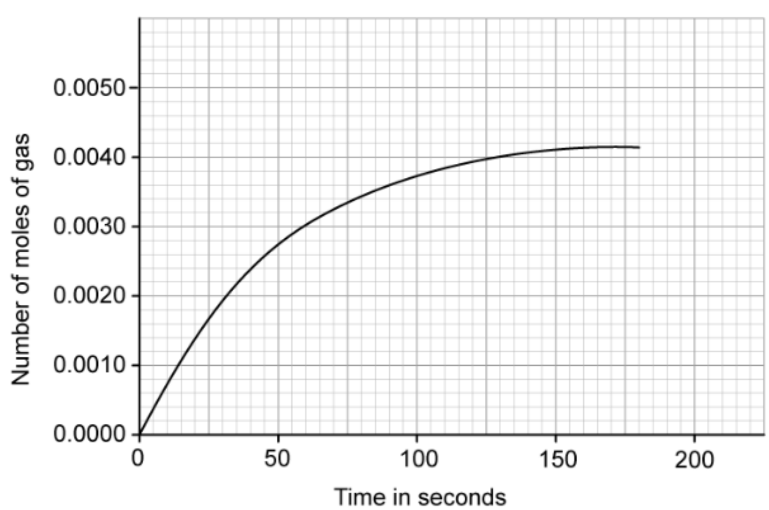
Time in seconds	Number of moles of gas
0	0.000
30	0.0012
60	0.0022
90	0.0030
120	0.0034
150	0.0037
180	0.0038

The student had already plotted the data for small calcium carbonate lumps.

Plot the data for the large calcium carbonate lumps and draw a line of best fit.

The student had already plotted the data for small calcium carbonate lumps.

Plot the data for the large calcium carbonate lumps and draw a line of best fit.



b) Determine the mean rate of reaction for **small** calcium carbonate lumps between 35 seconds and 90 seconds.

Give the unit.

Use the graph in part (a)

c) What conclusion can be made about the rate of reaction of small calcium carbonate lumps compared to large calcium carbonate lumps?

Give **one** reason for your answer.

d)

Complete and balance the equation for the reaction between calcium carbonate and hydrochloric acid.

_______ + _______ → $CaCl_2$ + _______ + _______

e)

State **one** advantage and **two** disadvantages of using a gas syringe as opposed to a upturned measuring cylinder when collecting gas.

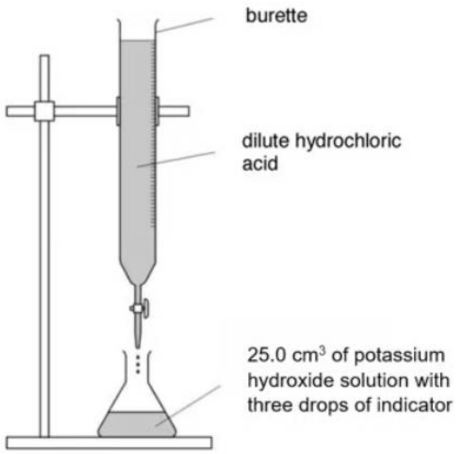
5) a)

A student does three titrations with dilute hydrochloric acid and potassium hydroxide solution.

Complete the equation to show the products formed during this reaction.

potassium hydroxide + hydrochloric acid → potassium ______ + _______

b)



Some of the apparatus the student uses is shown below.

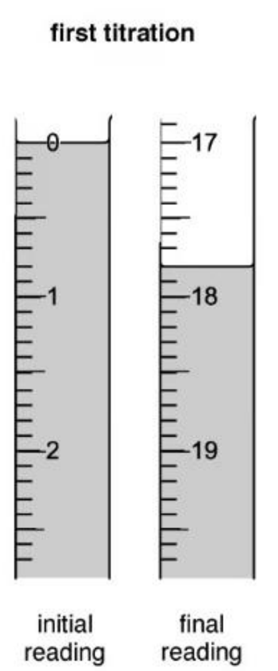
c) In her first titration the student measures the initial volume of hydrochloric acid in the burette.

She slowly adds the acid until the potassium hydroxide is just neutralised.

She then measures the volume of the hydrochloric acid again.

Describe how she can tell when the potassium hydroxide solution is just neutralised.

d) Look at the diagrams. They show parts of the burette during the first titration.



Here is the student's results table.

Titration number	1	2	3
final reading in cm^3		37.5	32.1
initial reading in cm^3		20.4	15.0
titre (volume of acid added) in cm^3		17.1	17.1

Complete the table by recording the burette readings from the diagrams.

6) a)

The following paragraph was taken from a student's notebook.

To make potassium chloride

25.0 cm^3 of aqueous potassium hydroxide were placed in a flask and a few drops of indicator were added. Dilute hydrochloric acid was added to the flask until the indicator changed colour. The volume of acid used was 19.0 cm^3.

What piece of apparatus should be used to measure the aqueous potassium hydroxide?

b)

i) Name a suitable indicator that could be used.

ii) The indicator would change from .... to ....

c)

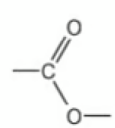
Which solution was more concentrated? Explain your answer.

d)

How could pure crystals of potassium chloride be obtained from this experiment?

7)

The ester linkage showing all the bonds is drawn as



or more simply it can be written as –COO–.

i) Give the structural formula of the ester ethyl ethanoate.

ii) Deduce the name of the ester formed from methanoic acid and butanol.

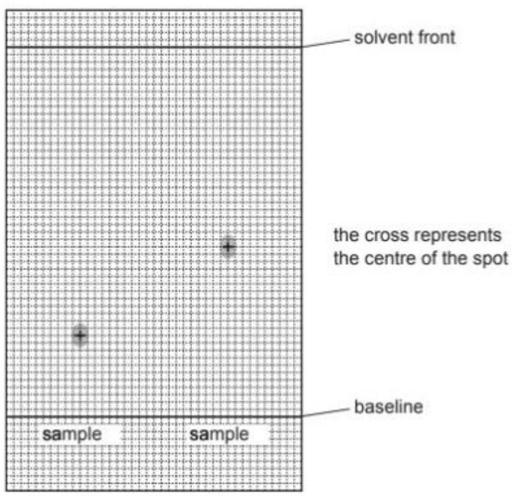
b) Draw the structural formula of the polyester formed from the following monomers.

$HOOCC_6H_4COOH$ and $HOCH_2CH_2OH$

You are advised to use the simpler form of the ester linkage.

c)

Esters can be used as solvents in chromatography. The following shows a chromatogram of plant acids.



An ester was used as the solvent and the chromatogram was sprayed with bromothymol blue.

i) Suggest why it was necessary to spray the chromatogram.

[2]

ii) Explain what is meant by the R_f value of a sample.

[1]

iii) Calculate the R_f values of the two samples and use the data in the table to identify the plant acids.

Plant acid	**R_f value**
tartaric acid	0.22
citric acid	0.30
oxalic acid	0.36
malic acid	0.46
succinic acid	0.60

8) a)

Describe how to separate the following. In each example, give a description of the procedure used and explain why this method works.

Copper powder from a mixture containing copper and zinc powders.

procedure..

explanation ....

b)

Nitrogen from a mixture of nitrogen and oxygen.

procedure..

explanation.....................................

c)

Glycine from a mixture of the two amino acids glycine and alanine. Glycine has the lower R_f value.

procedure......................................

explanation...................................

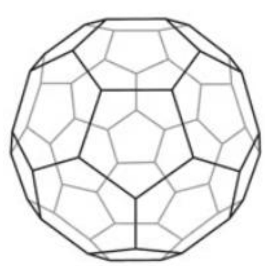
d) Magnesium hydroxide from a mixture of magnesium hydroxide and zinc hydroxide.

procedure..

explanation..

9)

In 1985 the fullerenes were discovered. They are solid forms of the element carbon. The structure of the C_{60} fullerene is given below.



i) In the C_{60} fullerene, how many other carbon atoms is each carbon atom bonded to?

ii) Another fullerene has a relative molecular mass of 840.
How many carbon atoms are there in one molecule of this fullerene?

b)

Fullerenes are soluble in liquid hydrocarbons such as octane. The other solid forms of carbon are insoluble.

Describe how you could obtain crystals of fullerenes from soot which is a mixture of fullerenes and other solid forms of carbon.

c)

A mixture of a fullerene and potassium is an excellent conductor of electricity.

i) Which other form of solid carbon is a good conductor of electricity?

[1]

ii) Explain why metals, such as potassium, are good conductors of electricity.

[2]

iii) The mixture of fullerene and potassium has to be stored out of contact with air. There are substances in unpolluted air which will react with potassium.
Name **two** potassium compounds which could be formed when potassium is exposed to air.

[2]

10)

Many compounds and elements have important uses.

Complete the table to show the name, formula and use of each compound and element.

name of compound or element	number of atoms in the formula	formula	use
chlorine	chlorine = 2	Cl_2	
	carbon = 1 hydrogen = 4	CH_4	
calcium carbonate	calcium = 1 carbon = 1 oxygen = 3		