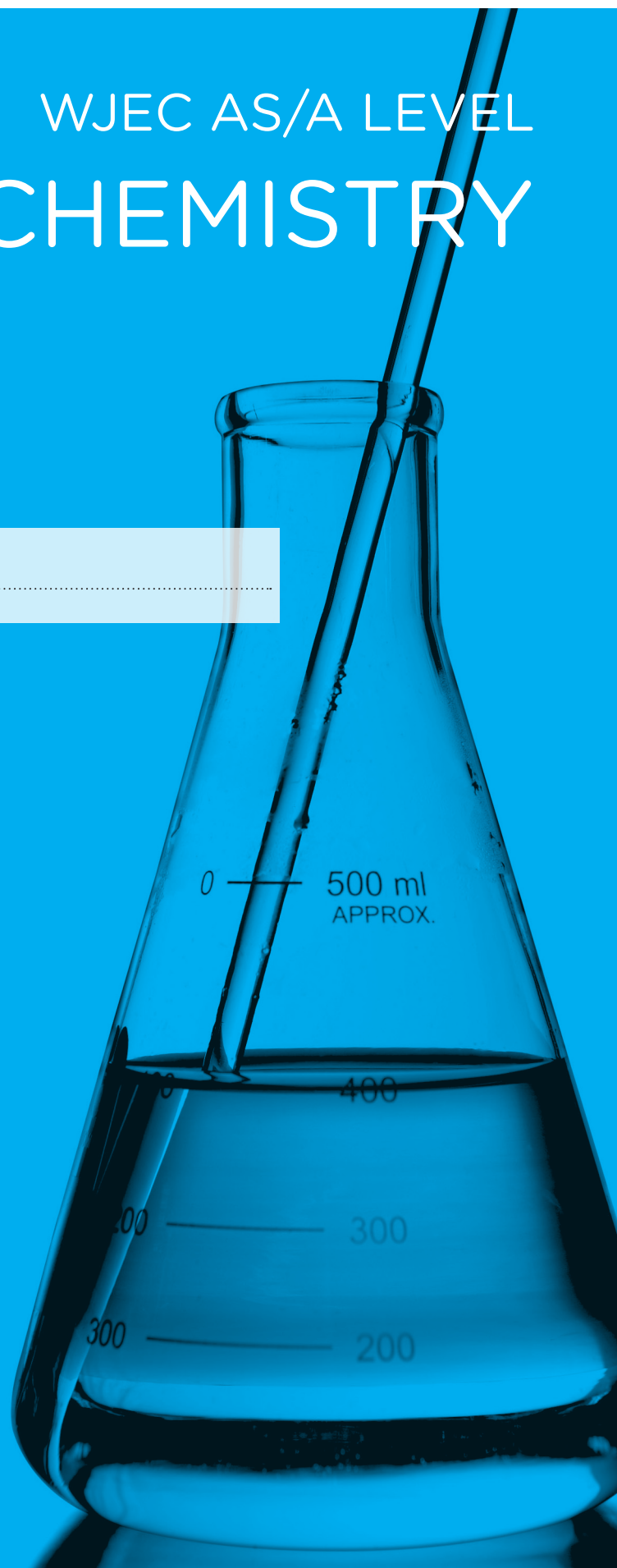


WJEC AS/A LEVEL CHEMISTRY

LAB BOOK

Name:



Topic	Specified practical work	Date completed	Teacher / Student comments
The Periodic Table	Gravimetric analysis		
	Identification of unknown solutions by qualitative analysis		
Simple equilibria and acid-base reactions	Preparation of a soluble salt by titration		
	Standardisation of an acid solution		
	Back titration		
	Double titration		
Thermochemistry	Indirect determination of an enthalpy change of reaction		
	Determination of an enthalpy change of combustion		
	Investigation of a rate of reaction by a gas collection method		
Rates of reaction	Study of an 'iodine clock' reaction		
Halogenoalkanes	Nucleophilic substitution reaction		
Alcohols and carboxylic acids	Preparation of an ester and separation by distillation		
Redox and standard electrode potential	Construction of electrochemical cells and measurement of E_{cell}		

Redox reactions	Simple redox titration		
	Estimation of copper in copper(II) salts		
Chemical kinetics	Determination of the order of a reaction		
	Determination of an equilibrium constant		
Equilibrium constants	Titration using a pH probe		
	Synthesis of a liquid organic product		
Acid-base equilibria	Synthesis of a solid organic product		
	Two-step synthesis		
	Planning a sequence of tests to identify organic compounds		
	Paper chromatography, including two-way separation		

Introduction

This lab book contains details relating to each of the 24 practical tasks specified as part of the content of WJEC GCE A level Chemistry. Detailed instructions are given for most tasks but some of them require an element of planning. In these cases, basic details are given and you must, for example, plan your own concentration ranges for a rates experiment or plan a method to identify unknown compounds.

Centres are free to amend these tasks or include preferred exercises provided that the same practical skills are developed, for example, any soluble salt can be prepared by titration.

Following completion of the specified practical tasks you should be able to

- apply investigative approaches and methods to practical work and think independently when undertaking practical work
- use a wide range of experimental and practical instruments, equipment and techniques appropriate to the knowledge and understanding included in the specification

Guidance notes

Methods of data collection and analysis

You should be able to

- describe with the aid of a clearly labelled diagram, the arrangement of apparatus for the experiment and the procedures to be followed
- describe how the data should be used in order to solve a problem or reach a conclusion
- set up apparatus correctly without assistance and follow instructions given
- undertake and record trial readings to determine the suitability of ranges and intervals where appropriate
- take repeat readings where appropriate
- make and record accurate measurements

Risk assessment

You should be able to assess the risks of your experiment.

Hazard	Risk	Control measure

Hazard – an object or chemical + the nature of the hazard

Risk – an ‘action’ in the method that can create a risk from a hazard

Control measure – must be practicable in the context of the practical

You will be required to produce your own risk assessment for some exercises. When you are not asked to do so centres must ensure that appropriate risk assessments are carried out in advance of all practical work.

Each exercise includes details of the hazards associated with the chemicals used. This does not amount to an appropriate risk assessment.

Table of results

You should be able to

- present numerical data and values in a single clear table of results
- use column headings for both quantity and unit e.g. Volume HCl / cm³
- include columns for all the primary data and values calculated from them
- record primary data to the same number of decimal places as the apparatus resolution e.g. if volume is measured to the nearest 0.05 cm³ then all volumes in the column should be recorded to the nearest 0.05 cm³

Recording readings and significant figures

All primary data should be recorded to the resolution of the apparatus used. Any data calculated from the primary data should be given to the same number of significant figures (or a maximum of one extra) as the primary data. The number of significant figures should be consistent within a column of data.

Graphs

You should be able to

- include a title and axes which are labelled with scales and units
- make sure the scales are convenient to use, so that readings may easily be taken from the graph – avoid scales which use factors of 3 – and that the plotted points occupy at least half of both the vertical and horizontal extent of the graph grid
- consider carefully whether your plotted points suggest a straight line or a curve, then draw in your best fit line either with the aid of a ruler or (if a curve) by a freehand sketch
- determine the gradient of a graph, showing clearly the readings you use by drawing a right angled triangle (this should be large so that accuracy is preserved)

Estimating uncertainties

You should be able to

- identify the measurement which involves the greatest uncertainty (based on apparatus resolution), likely to be a volume or temperature
- express the uncertainty (single value and difference between two readings) as a percentage of the measured value
- express the result to a sensible number of significant figures (4 sig figs for 0.1%; 3 sig figs for 1% and 2 sig figs for 10%)

Conclusions and evaluations

You should be able to

- use data to solve a problem or reach a conclusion
- evaluate experimental methods and suggest improvements

GRAVIMETRIC ANALYSIS

Specification reference: AS Unit 1.6

Aim

Identification of an unknown metal chloride

Apparatus and chemicals

- Access to balance – ideally 3 decimal place (minimum 2 decimal place)
- Access to drying oven

- 2 x 250 cm³ beaker
- 2 x 100 cm³ beaker
- spatula
- 10 cm³ measuring cylinder
- 3 x dropping pipette
- Bunsen burner, tripod and gauze or electric hotplate
- filter funnel
- filter paper
- wash bottle

- sample of unknown chloride
- deionised water
- 0.5 mol dm⁻³ AgNO₃ solution
- 6 mol dm⁻³ HNO₃ solution
- 2 mol dm⁻³ HCl solution
- wash solution – 4 cm³ of 6 mol dm⁻³ HNO₃ per dm³ deionised water

Safety considerations

0.5 mol dm ⁻³ AgNO ₃ solution	– corrosive
6 mol dm ⁻³ HNO ₃ solution	– corrosive
2 mol dm ⁻³ HCl solution	– irritant

Method

1. Accurately weigh around 0.3 g of the unknown chloride and transfer to a 250 cm³ beaker.
2. Dissolve the sample in approximately 100 cm³ of deionised water and add 3 cm³ of 6 mol dm⁻³ HNO₃.
3. Slowly add 20 cm³ of AgNO₃ solution until silver chloride is seen to coagulate, then add a further 5 cm³ of AgNO₃.
4. Heat the beaker carefully for 10 minutes without allowing the solution to boil.
5. Leave the solution to cool for at least 2 hours.
6. Accurately weigh a filter paper and place in a filter funnel.
7. Decant the liquid from the beaker into the funnel.
8. Wash the precipitate in the beaker with 3 cm³ of wash solution.
9. Transfer all the precipitate to the filter funnel and wash with 20 cm³ of wash solution.
10. Test the used wash solution by adding 3 drops of HCl solution. If a precipitate forms (indicating the presence of Ag⁺ ions), wash with a further 10 cm³ and re-test.
11. Dry the precipitate in the filter paper at 105 °C until constant mass is attained.

IDENTIFICATION OF UNKNOWN SOLUTIONS BY QUALITATIVE ANALYSIS

Specification reference: AS Unit 1.6

Aim

To plan and carry out a method to identify six inorganic salts by the interactions between their solutions

Apparatus and chemicals

- 20 x test tube
- test tube rack
- 5 x dropping pipette

- solutions of the following salts, randomly labelled A–F
 - $\text{Ba}(\text{NO}_3)_2$
 - $\text{Pb}(\text{NO}_3)_2$
 - MgSO_4
 - KI
 - Na_2CO_3
 - $\text{Zn}(\text{NO}_3)_2$

Safety considerations

$\text{Ba}(\text{NO}_3)_2$ - toxic
 $\text{Pb}(\text{NO}_3)_2$ - toxic



Planning

1. Construct and complete a table to show the expected observations when each solution interacts with the other five.
2. Record a summary of the observations expected for each salt, e.g. two white precipitates, one yellow precipitate and two 'no change'.
3. Construct another similar table but this time labelled with the letters A–F rather than the names of the salts.

Method

1. Test approximately 2 cm^3 of each solution with a few drops of each of the other solutions in turn and record your observations in the second table.

PREPARATION OF A SOLUBLE SALT BY TITRATION

Specification reference: AS Unit 1.7

Aim

To prepare crystals of sodium chloride by using titration followed by evaporation

Apparatus and chemicals

- 50 cm³ burette
- funnel
- 25 cm³ pipette and filler
- 100 cm³ conical flasks
- evaporating basin

- 0.1 mol dm⁻³ NaOH solution
- 0.1 mol dm⁻³ HCl solution
- phenolphthalein indicator

Safety considerations

0.1 mol dm ⁻³ NaOH solution	- irritant
0.1 mol dm ⁻³ HCl solution	- irritant
phenolphthalein indicator	- flammable

Method

1. Using a pipette, measure 25.00 cm^3 of NaOH solution and pour it into a conical flask.
2. Add two drops of phenolphthalein.
3. Pour the HCl solution into a burette.
4. Record the initial volume of HCl solution.
5. Add the hydrochloric acid from the burette into the conical flask a little at the time while swirling the conical flask.
6. When the phenolphthalein starts to turn from pink to colourless, add the HCl solution a drop at the time until one drop is sufficient to turn the solution colourless. Record the volume of HCl solution needed.
7. Carry out the titration again using 25.00 cm^3 of NaOH solution and exactly the same volume of HCl solution.
8. Do not add indicator this time.
9. Gently heat the solution from the conical flask in an evaporating basin until its volume decreases by around a half.
10. Leave the evaporating basin to cool allowing crystals to form.

STANDARDISATION OF AN ACID SOLUTION

Specification reference: AS Unit 1.7

Aim

To find the concentration of hydrochloric acid using a standard solution of anhydrous sodium carbonate

Apparatus and chemicals

Part 1

- access to a 3 decimal place digital balance (minimum 2 decimal place)
- access to deionised water
- weighing bottle/boat
- 250 cm³ volumetric flask and funnel
- 250 cm³ beaker and glass rod
- labels
- wash bottle containing distilled water
- Pasteur pipette
- anhydrous Na₂CO₃

Part 2

- 50cm³ burette and funnel
- burette clamp and stand
- 25 cm³ bulb/volumetric pipette with safety filler
- 2 x 250 cm³ conical flask
- white tile
- 0.2 mol dm⁻³ HCl solution
- methyl orange indicator

Safety considerations

Na₂CO₃

0.2 mol dm⁻³ HCl solution

- irritant

- irritant



Method

Part 1 – Making up the standard solution

1. Accurately weigh out approximately 2.75 g of anhydrous Na_2CO_3 into a weighing bottle.
2. Record the mass with the correct number of significant figures.
3. Tip the solid into a 250 cm^3 beaker and reweigh the weighing bottle and traces.
4. Dissolve the solid in deionised water, stirring with a glass rod. Ensure you do not add more than 150 cm^3 of distilled water at this stage.
5. Pour the solution into the 250 cm^3 volumetric flask via a funnel.
6. Rinse the beaker and glass rod at least three times, transfer the washings into the flask each time.
7. Make the solution up to the mark with distilled water so that the bottom of the meniscus is level with the graduation mark. The flask and graduation mark must be at eye level when the final drops are added using a Pasteur pipette.
8. Add the stopper and shake the mixture thoroughly.
9. Label the volumetric flask ready for use during the second part of the experiment.

Part 2 – Titrating the standard solution with hydrochloric acid

1. Using a funnel, pour a small volume of the HCl solution into the burette. Rinse the burette with this solution to remove any water left behind after washing.
2. Now fill the burette with the HCl solution and record the initial burette reading.
3. Rinse the volumetric pipette with a small volume of sodium carbonate solution.
4. Accurately pipette 25.0 cm^3 of the Na_2CO_3 solution into a conical flask add 3 drops of methyl orange indicator.
5. The standard solution is titrated with the acid until on addition of one drop of acid the indicator changes colour from yellow to pink/red. Use the white tile to help you see the colour change.
6. Record the burette reading and repeat until the results are concordant.

BACK TITRATION

Specification reference: AS Unit 1.7

Aim

Determination of the percentage of calcium carbonate in limestone

Apparatus and chemicals

- access to a 3 decimal place digital balance (minimum 2 decimal place)
- 25 cm³ bulb/volumetric pipette with safety filler
- pestle and mortar
- 250 cm³ conical flask
- hot plate/hot water bath/Bunsen burner
- 50cm³ burette and funnel
- burette clamp and stand
- 250 cm³ conical flask
- white tile
- limestone chips (or antacid tablets)
- 0.1 mol dm⁻³ NaOH solution
- 0.5 mol dm⁻³ HCl solution
- phenolphthalein indicator

Safety considerations

0.1 mol dm⁻³ NaOH solution

0.5 mol dm⁻³ HCl solution

phenolphthalein indicator

- irritant

- irritant

- flammable



Procedure

1. Using a pestle and mortar, grind the limestone chips (or antacid tablet) into a fine powder.
2. Accurately weight approximately 0.5 g of limestone powder and transfer to a conical flask.
3. Record the mass of limestone added.
4. Using a 25 cm³ pipette, add 25 cm³ HCl solution.
5. Stir the reaction mixture vigorously with a stirring rod and heat gently with regular stirring.
6. When the limestone has dissolved completely, cool to room temperature.
7. Add a few drops of phenolphthalein indicator and titrate against the standardised NaOH solution.
8. Record the volume of NaOH solution needed to neutralise the excess HCl solution and use this to calculate the percentage CaCO₃ in limestone.

DOUBLE TITRATION

Specification reference: AS Unit 1.7

Aim

To determine the concentration and the mass of sodium hydroxide and sodium carbonate in a mixed solution

Apparatus and chemicals

- 50cm³ burette and funnel
- burette clamp and stand
- 25 cm³ bulb/volumetric pipette with safety filler
- 2 x 250 cm³ conical flask
- white tile

- 0.1 mol dm⁻³ HCl solution
- NaOH/Na₂CO₃ mixed solution
- phenolphthalein indicator
- methyl orange indicator

Safety considerations

mixed NaOH/Na ₂ CO ₃ solution	- irritant
0.1 mol dm ⁻³ HCl solution	- irritant
phenolphthalein indicator	- flammable
methyl orange indicator	- flammable

Method

1. Titrate a 25.00 cm³ sample of the mixed solution against HCl solution using phenolphthalein as the indicator. Do not agitate the flask any more than the minimum necessary to mix the acid.
2. Record the volume used at the phenolphthalein end-point.
3. Add methyl orange and continue titrating until its end-point.
4. Record the total volume of hydrochloric acid added to this end-point.
5. Repeat as necessary until the titration values obtained agree within 0.20 cm³ and separately average results for the two values.
6. Calculate the concentration of NaOH and Na₂CO₃ in a mixed solution and then the mass in grams.

INDIRECT DETERMINATION OF AN ENTHALPY CHANGE OF REACTION

Specification reference: AS Unit 2.1

Aim

Indirect determination of the enthalpy change of reaction of magnesium oxide and carbon dioxide to form magnesium carbonate

Apparatus and chemicals

- access to a 3 decimal place digital balance (minimum 2 decimal place)
- thermometer
- simple calorimeter
- 25 cm³ bulb/volumetric pipette with safety filler
- stopwatch
- spatula
- weighing boat

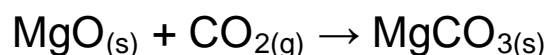
- 2 mol dm⁻³ HCl solution
- dry MgO
- dry MgCO₃

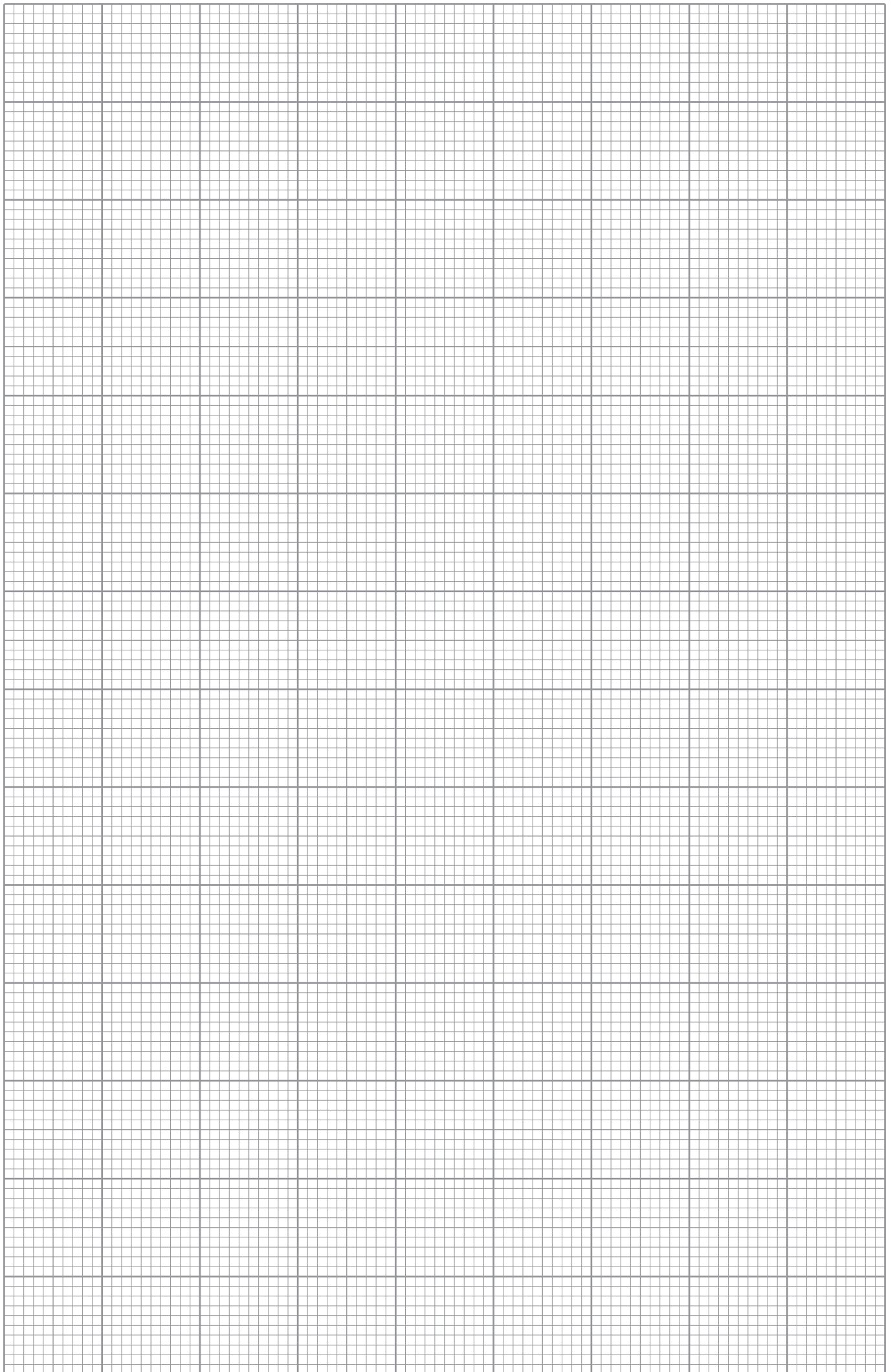
Safety considerations

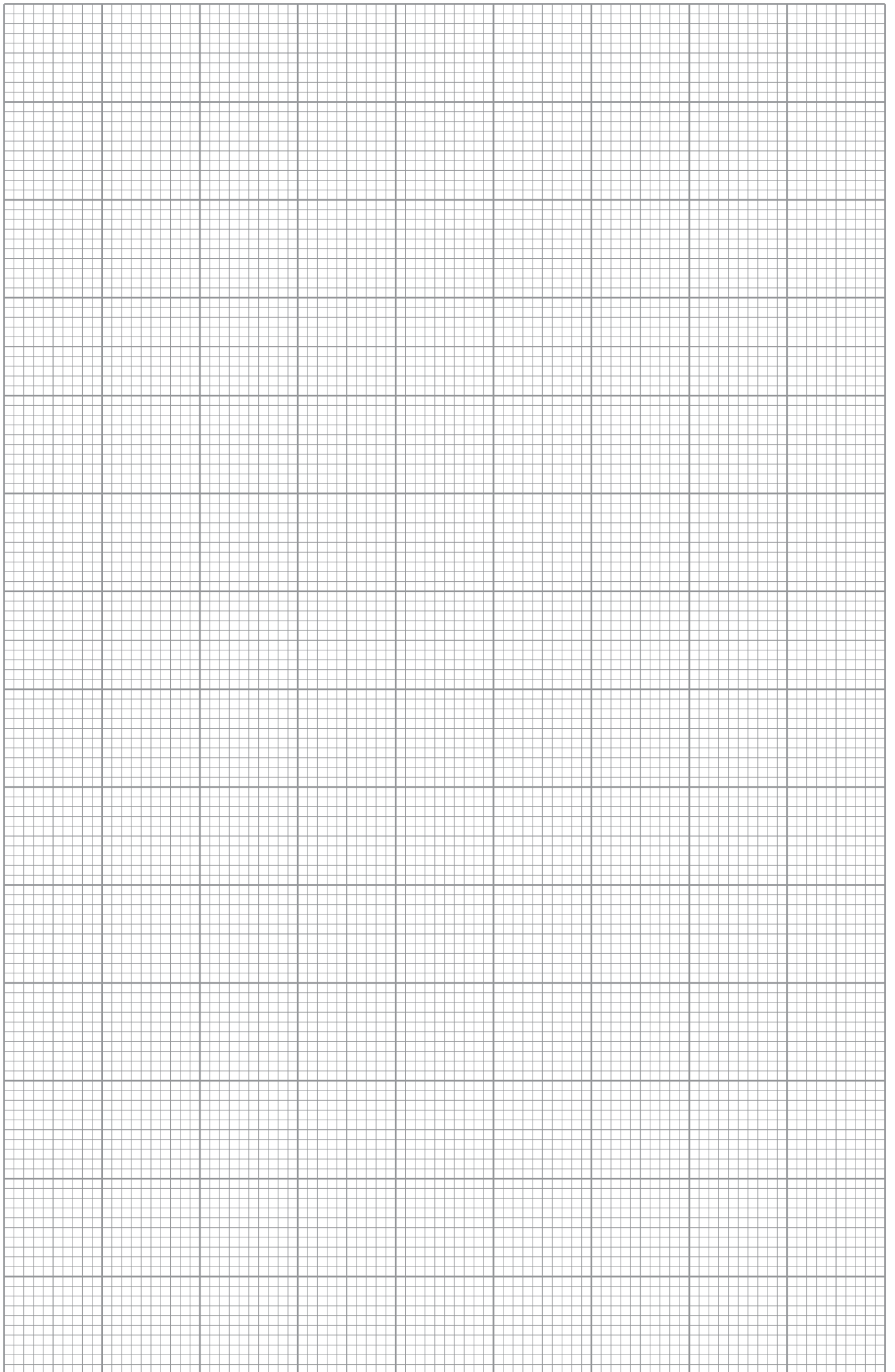
2 mol dm⁻³ HCl solution - irritant

Method

1. Collect the equipment listed above.
2. Measure 50 cm³ of HCl solution into the calorimeter using the pipette to measure out 2 measurements of 25 cm³.
3. Place the thermometer into the HCl solution and leave it in to allow the temperature reading to stabilise.
4. Accurately weigh out approximately 0.90 g of MgO in a weighing boat.
5. Accurately weigh the weighing boat and MgO. Record the mass.
6. Record the temperature of the acid. At the same time, start the stopwatch.
7. Record the temperature of the acid every 30 seconds for 2 minutes and 30 seconds.
8. When the stopwatch reaches 3 minutes, add the MgO to the HCl solution and mix it together thoroughly. Keep the bulb of the thermometer submerged in the reaction mixture.
9. When the stop watch reaches 3 minutes 30 seconds, record the temperature of the reaction mixture.
10. Continue to record the temperature of the reaction mixture every 30 seconds until the temperature drops for 5 consecutive readings.
11. Weigh the weighing boat again. Record the mass. Use this mass and the mass obtained in step 5 to calculate the mass of MgO added to the calorimeter.
12. Use the data to construct an appropriate graph and calculate the enthalpy change of the reaction between MgO and HCl.
13. Repeat steps 2 – 12 using approximately 3.5 g of MgCO₃.
14. Use the values of ΔH obtained for each reaction to calculate the enthalpy change for the following reaction:







DETERMINATION OF AN ENTHALPY CHANGE OF COMBUSTION

Specification reference: AS Unit 2.1

Aim

Determination of the enthalpy change of combustion of methanol and ethanol

Apparatus and chemicals

- access to deionised water
- access to a 3 decimal place digital balance (minimum 2 decimal place)

- clamp stand
- 250cm³ conical flask
- spirit burner
- heatproof mat
- thermometer

- CH₃OH
- CH₃CH₂OH

Safety considerations

CH₃CH₂OH - highly flammable
 CH₃OH - highly flammable, toxic



Method

1. Add 100 cm³ of deionised water to a 250 cm³ conical flask.
2. Clamp the conical flask to a stand at a suitable height so that the spirit burner can be placed below it.
3. Weigh a spirit burner and lid containing methanol using a mass balance and record the mass.
4. Record the initial temperature of the water using the thermometer.
5. Place the spirit burner under the conical flask and light the wick.
6. Allow the flame to heat the water by around 40 °C.
7. Replace the cap to extinguish the flame.
8. Record the final temperature of the water.
9. Re-weigh the spirit burner and cap and record in your table.
10. Calculate the mass of alcohol used.
11. Repeat steps 1-10 for ethanol.
12. Determine the energy released by each alcohol and calculate the enthalpy change of combustion - $\Delta_c H^\circ$.

INVESTIGATION OF A RATE OF REACTION BY A GAS COLLECTION METHOD

Specification reference: AS Unit 2.2

Aim

Determination of the rate of reaction of hydrochloric acid and calcium carbonate

Apparatus and chemicals

- access to a 3 decimal place digital balance (minimum 2 decimal place)
- safety goggles
- 250 cm³ conical flask
- 100 cm³ gas syringe with delivery tube and rubber stopper
- 50 cm³ measuring cylinder
- weighing boat
- spatula
- stopwatch
- clamp and stand

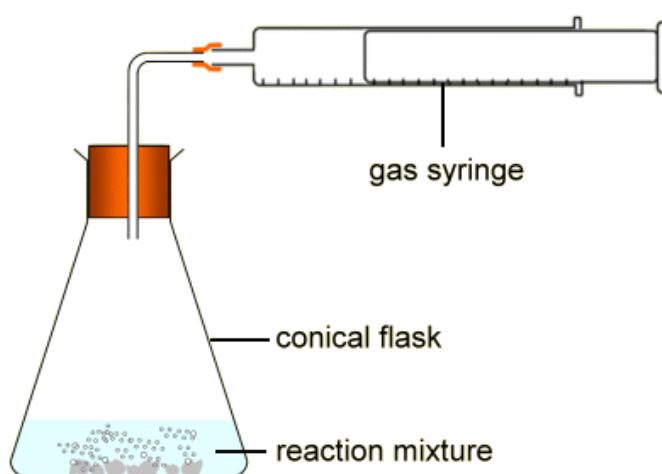
- HCl solutions of different concentrations
- CaCO₃ powder

Safety considerations

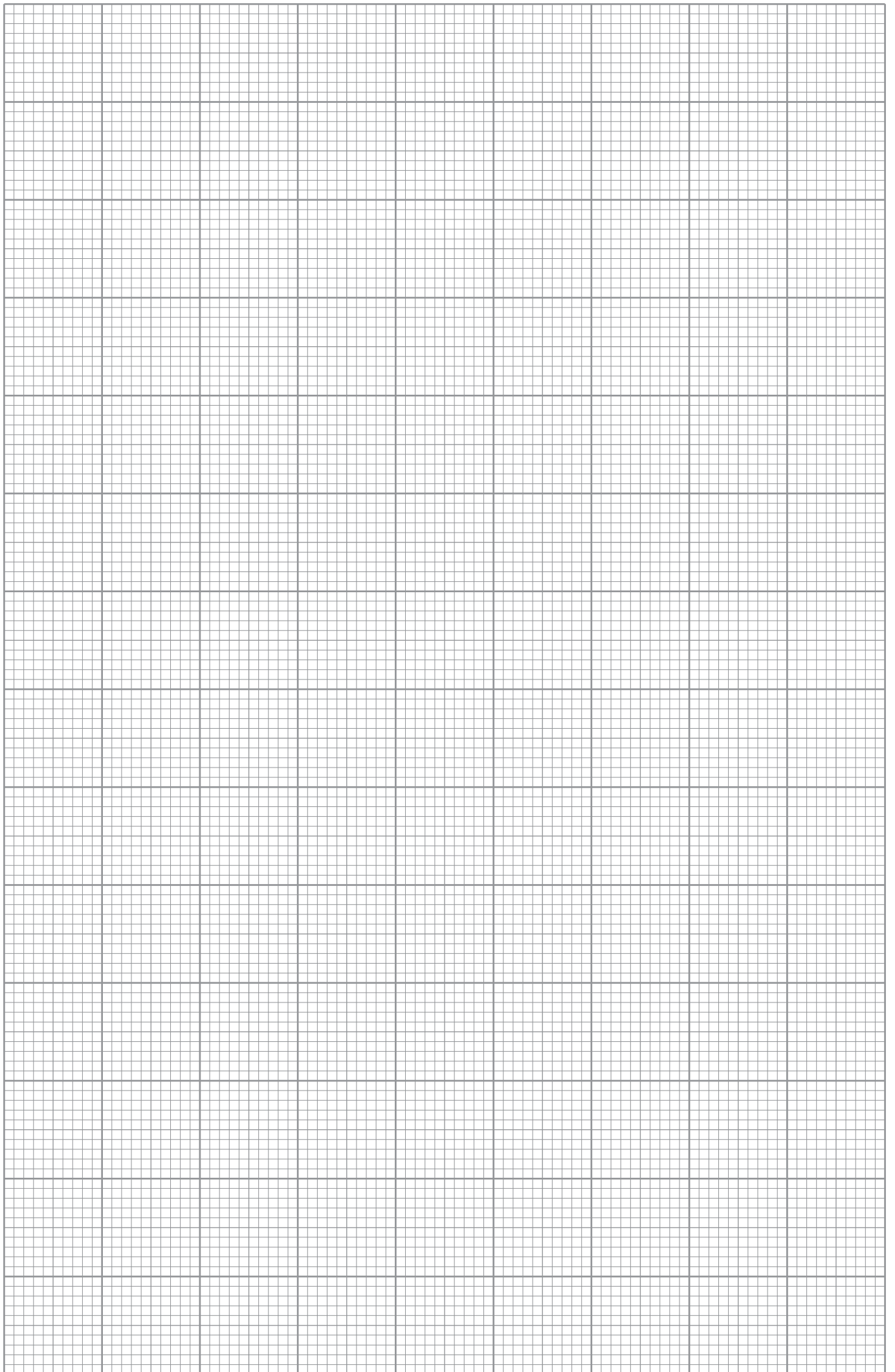
HCl solution - irritant

Method

1. Measure 50 cm^3 of HCl solution of known concentration into the conical flask using the 50 cm^3 measuring cylinder.
2. Measure out 2 grams of CaCO_3 powder using a weighing boat.
3. Set up the gas syringe in a clamp and stand.
4. Put the CaCO_3 powder into the conical flask and then quickly put the rubber stopper and delivery tube into the neck of the conical flask. At the same time, start the stopwatch.



6. Stop the stopwatch as soon as the volume of gas in the syringe reaches 100 cm^3 .
7. Record this result in an appropriate table and calculate the average rate for this reaction in cm^3s^{-1} .
8. Repeat steps 2-7 using a different concentration of HCl solution.
9. Use your data to plot a graph of rate ($1/t$) against concentration of HCl solution.



STUDY OF AN 'IODINE CLOCK' REACTION

Specification reference: AS Unit 2.2

Aim

To study the kinetics of the oxidation of iodide ions by hydrogen peroxide in acid solution

Apparatus and chemicals

- access to deionised water
- stopwatch
- 4 x 10 cm³ measuring cylinders
- 25 cm³ bulb/volumetric pipette with safety filler
- 50cm³ burette and funnel
- burette clamp and stand
- 5 x 250 cm³ conical flasks

- 0.1 mol dm⁻³ H₂O₂ solution
- 1 mol dm⁻³ H₂SO₄ solution
- 0.1 mol dm⁻³ KI solution
- 0.005 mol dm⁻³ Na₂S₂O₃
- starch solution

Safety considerations

1 mol dm⁻³ H₂SO₄ solution - irritant

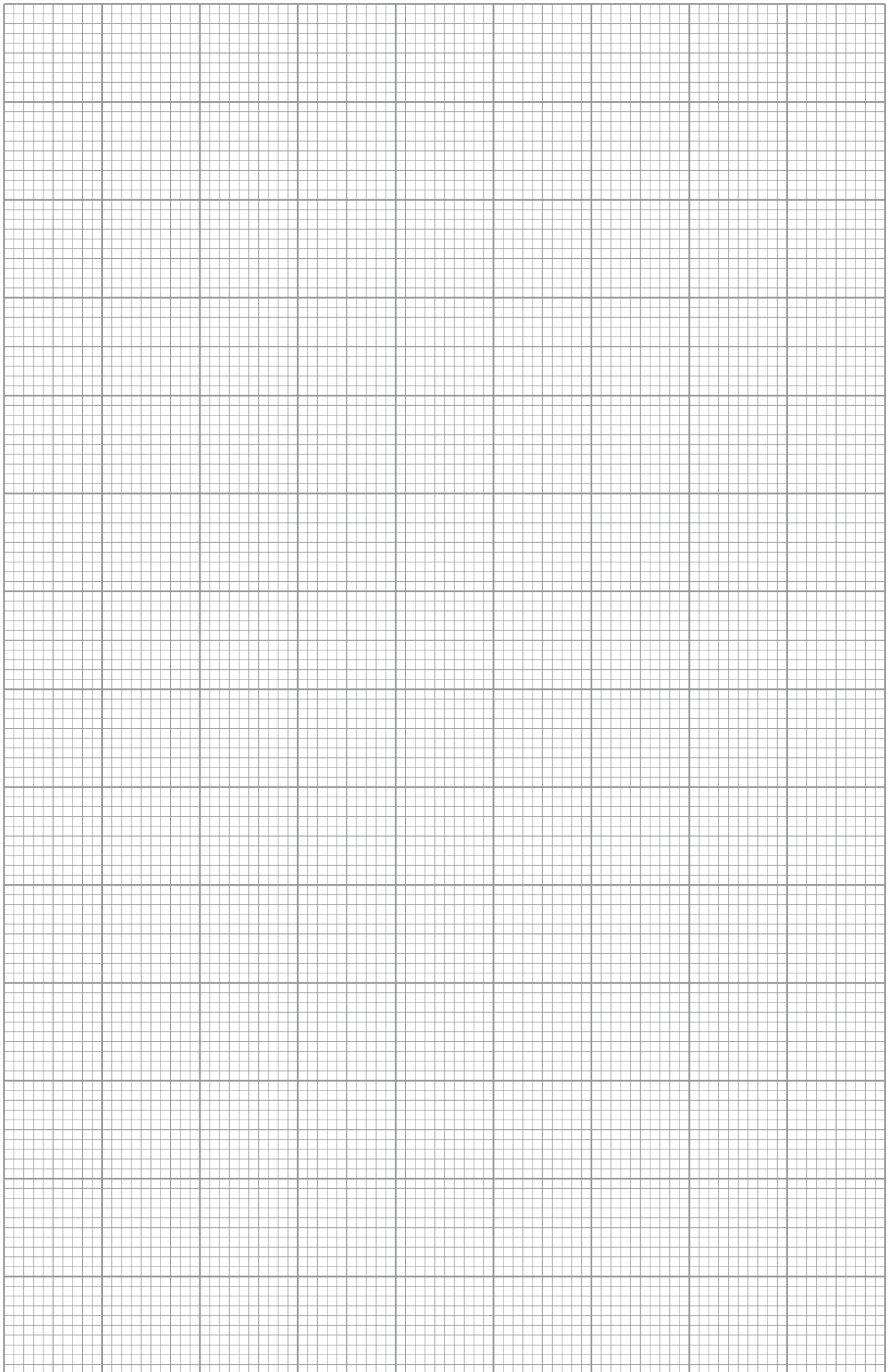
Planning

1. Decide what volumes of H_2O_2 solution and deionised water you will mix together to get at least 5 different concentrations of H_2O_2 . The total volume must not exceed 5 cm^3 .

General method

1. In separate conical flasks, make up the solutions according to the table below. Do not add the hydrogen peroxide yet.
2. Add 1 cm^3 starch solution to each flask and mix thoroughly.
3. Rapidly add the hydrogen peroxide to flask 1, starting the stopwatch immediately after this addition.
4. Swirl to mix the reaction mixture thoroughly.
5. Stop timing when the solution turns blue-black and record the time.
6. Repeat the experiment for flasks 2-5.

Flask	Volume H_2SO_4 (cm^3)	Volume $\text{Na}_2\text{S}_2\text{O}_3$ (cm^3)	Volume KI (cm^3)	Volume H_2O (cm^3)	Volume H_2O_2 (cm^3)
1	10	10	25		
2	10	10	25		
3	10	10	25		
4	10	10	25		
5	10	10	25		



NUCLEOPHILIC SUBSTITUTION REACTION

Specification reference: AS Unit 2.6

Aim

To prepare a pure sample of butan-1-ol from 1-bromobutane and sodium hydroxide

Apparatus and chemicals

- 100 cm³ round bottom flask
- 100 cm³ beaker
- 100 cm³ conical flask
- 25 cm³ measuring cylinder
- reflux condenser
- thermometer
- specimen tube
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand

- 0.1 mol dm⁻³ NaOH solution
- CH₃CH₂CH₂CH₂Br

Safety considerations

CH₃CH₂CH₂CH₂Br
0.1 mol dm⁻³ NaOH solution

- flammable, irritant
- irritant



Method

1. Measure 25 cm³ of CH₃CH₂CH₂CH₂Br using a measuring cylinder and decant to a round bottomed flask. This step should be performed in the fume cupboard.
2. Add 25 cm³ of 0.1 mol dm⁻³ NaOH solution and a few anti-bumping granules in to the round bottom flask. This step should be performed in the fume cupboard.
3. Swirl the flask gently to mix the reagents and set up the reflux condenser apparatus for refluxing (diagram 1).
4. Turn on the water to run through the condenser.
5. Turn on the heating mantle (or heat the water bath with a Bunsen burner) and heat the solution to reflux for 15 minutes.
6. Rearrange the apparatus for distillation (diagram 2).
7. Distil off the CH₃CH₂CH₂CH₂OH produced and collect in a clean, dry 100 cm³ conical flask.
8. Record the temperature at which the liquid product is collected.

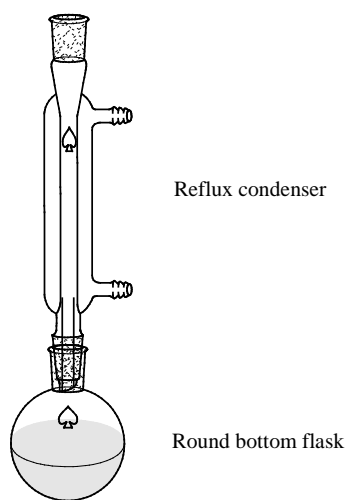


diagram 1

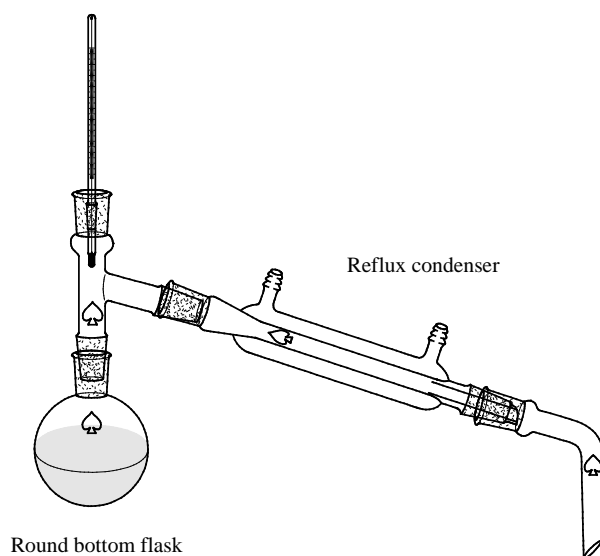


diagram 2

Boiling point of butan-1-ol	
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PREPARATION OF AN ESTER AND SEPARATION BY DISTILLATION

Specification reference: AS Unit 2.7

Aim

To prepare a pure sample of ethyl ethanoate from ethanoic acid and ethanol

Apparatus and chemicals

- 100 cm³ round bottom flask
- 100 cm³ beaker
- 100 cm³ conical flask
- 10 cm³ measuring cylinder
- 25 cm³ measuring cylinder
- reflux condenser
- thermometer
- specimen tube
- labels/suitable pen
- anti-bumping granules
- warm water bath (approximately 50 °C)
- clamp stand

- CH₃CH₂OH
- CH₃COOH
- concentrated H₂SO₄ solution

Safety considerations

CH₃CH₂OH
 CH₃COOH
 H₂SO₄ solution

- flammable
 - irritant
 - corrosive



Method

1. Measure 25 cm³ of CH₃COOH using a measuring cylinder and decant to a round bottomed flask.
2. Add 10 cm³ of CH₃CH₂OH solution and a few anti-bumping granules in to the round bottom flask.
3. Add 10 drops of concentrated H₂SO₄ solution.
4. Swirl the flask gently to mix the reagents.
5. Warm the reaction mixture gently with the warm water bath for 15 minutes.
6. Set up the distillation apparatus (see diagram).
7. Distil off the CH₃COOCH₂CH₃ produced and collect in a clean, dry 100 cm³ conical flask.
8. Record the temperature at which the liquid product is collected.

Boiling point of ethyl ethanoate	
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CONSTRUCTION OF ELECTROCHEMICAL CELLS AND MEASUREMENT OF E_{CELL}

Specification reference: A2 Unit 3.1

Aim

Construction of a copper/zinc electrochemical cell and the determination of E_{cell}

Apparatus and chemicals

- wires
- crocodile clips
- voltmeter
- 2 x 100 cm³ beakers
- filter paper cut into a long strip

- Cu foil strip
- Zn foil strip
- 1 mol dm⁻³ CuSO₄ solution
- 1 mol dm⁻³ ZnSO₄ solution
- saturated KNO₃ solution

Safety considerations

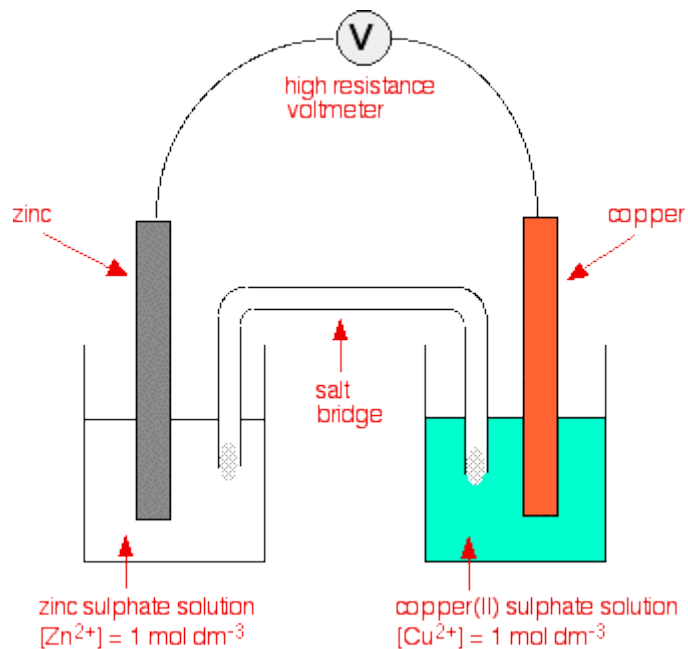
1 mol dm ⁻³ CuSO ₄ solution	- harmful, dangerous to environment
1 mol dm ⁻³ ZnSO ₄ solution	- irritant, dangerous to environment
Saturated KNO ₃ solution	- oxidising



Method

1. Measure 50 cm³ of CuSO₄ solution into one of the beakers.
2. Measure 50 cm³ of ZnSO₄ solution into the other beaker.
3. Place the copper foil in the CuSO₄ solution.
4. Place the zinc foil in the ZnSO₄ solution.
5. Connect the zinc foil strip and Cu foil strip to the ammeter.
6. Connect the voltmeter across the Zn foil and copper foil connections.
7. Place the two beakers directly next to each other.
8. Soak the filter paper in the saturated KNO₃ solution and place it across the two beakers.
9. Ensure that the ends of the filter paper are in direct contact with the solutions in the two beakers. This is called a salt bridge.
10. Measure the potential difference of the cell using the voltmeter.

Diagram



Potential difference of cell	
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SIMPLE REDOX TITRATION

Specification reference: A2 Unit 3.2

Aim

To determine the relative molecular mass of an iron(II) salt by titration with standard potassium manganate(VII) solution

Apparatus and chemicals

- access to 3 decimal place mass balance (minimum 2 decimal place)
- safety goggles
- a 50 cm³ burette and funnel
- 25 cm³ pipette and filler
- 250 cm³ conical flasks
- 250 cm³ volumetric flask
- unknown iron(II) salt
- 1.0 mol dm⁻³ H₂SO₄ solution
- standardised KMnO₄ solution (approximately 0.02 mol dm⁻³)

Safety considerations

1.0 mol dm⁻³ H₂SO₄ solution
KMnO₄ solution

- irritant
- harmful, oxidising



Method

1. Weigh out accurately, about 9.8 g of the iron(II) salt provided and record the mass.
2. Make the salt up to 250 cm³ of a standard solution in H₂SO₄ solution.
3. Titrate 25cm³ portions of this solution against the standardised KMnO₄ solution.
4. Use your results to calculate the relative molecular mass of the iron(II) salt.

ESTIMATION OF COPPER IN COPPER(II) SALTS

Specification reference: A2 Unit 3.2

Aim

To determine the percentage by mass of copper in copper(II) sulfate crystals

Apparatus and chemicals

- access to a 3 decimal place mass balance (minimum 2 decimal place)
- weighing boat
- 2 x 50 cm³ beakers
- 250 cm³ beaker
- 3 x 250 cm³ conical flask
- 250 cm³ volumetric flask with stopper
- 25 cm³ bulb pipette
- pipette filler
- burette, funnel and stand
- white tile
- wash bottle of de-ionised water
- 2 x Pasteur pipettes
- CuSO₄·5H₂O crystals
- 0.1 mol dm⁻³ Na₂S₂O₃ solution
- 0.2% starch solution
- KI powder

Safety considerations

CuSO₄·5H₂O crystals - harmful, dangerous to the environment
 KI powder - irritant



Method

1. Accurately weigh out about 6 g copper(II) sulfate crystals into a weighing boat.
2. Use the copper(II) sulfate crystals to make up 250 cm³ of standardised copper(II) sulfate solution.
3. Pipette 25 cm³ of this solution into a conical flask.
4. Add 1.5 g potassium iodide and swirl thoroughly.
5. Titrate this solution with standard 0.1 mol dm⁻³ Na₂S₂O₃ in a burette. When the iodine colour fades, add 1 cm³ starch indicator.
6. Use your titration data to calculate the percentage by mass of copper in the copper(II) sulfate crystals.

DETERMINATION OF THE ORDER OF A REACTION

Specification reference: A2 Unit 3.5

Aim

To determine of the order of reaction for the oxidation of iodide ions by hydrogen peroxide in acid solution

Apparatus and chemicals

- stopwatch
- 100 cm³ conical flask
- stirring rod
- 4 x 10 cm³ measuring cylinder
- 5 cm³ measuring cylinder
- 1 cm³ measuring cylinder

- 0.1 mol dm⁻³ H₂O₂ solution
- 1.0 mol dm⁻³ H₂SO₄ solution
- 0.1 mol dm⁻³ KI solution
- 0.005 mol dm⁻³ Na₂S₂O₃ solution
- starch solution

Safety considerations

H₂O₂ solution
H₂SO₄ solution

- harmful, oxidising
- irritant

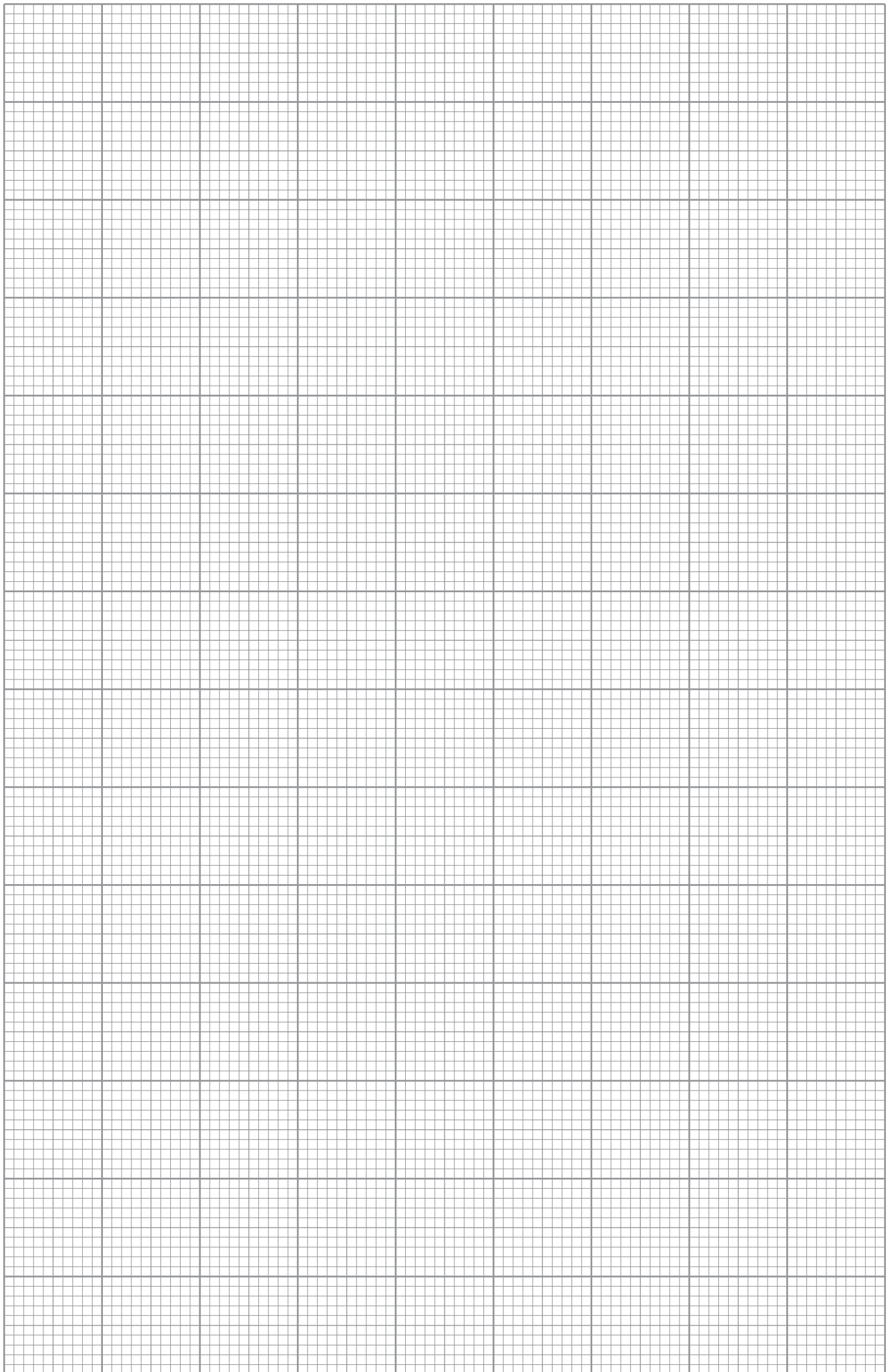


Planning

1. Decide what volumes of H_2O_2 solution and deionised water you will mix together to get at least 5 different concentrations of H_2O_2 . The total volume must not exceed 5 cm^3 .

General method

1. Prepare the reaction mixture by adding the following reagents to a 100 cm^3 conical flask.
 - 10.0 cm^3 H_2SO_4 solution
 - 10.0 cm^3 $\text{Na}_2\text{S}_2\text{O}_3$ solution
 - 15.0 cm^3 KI solution
 - 1.0 cm^3 starch solution
 - 9.0 cm^3 deionised water
2. Be ready with the stopwatch. Rapidly add 5.0 cm^3 of H_2O_2 solution to the reaction mixture and simultaneously start the stopwatch. Ensure the reaction mixture is thoroughly mixed.
3. Stop the watch immediately when the blue colour appears and record the time.
4. Repeat steps 1-3 using the other four concentrations of H_2O_2 solution.
5. Calculate the rate of reaction for each experiment.
6. Plot a graph of rate of reaction against $[\text{H}_2\text{O}_2]$ solution and use this to calculate the order of reaction with respect to $[\text{H}_2\text{O}_2]$.



DETERMINATION OF AN EQUILIBRIUM CONSTANT

Specification reference: A2 Unit 3.8

Aim

To determine the equilibrium constant for the reaction of ethanol with ethanoic acid

Apparatus and chemicals

- communal burette of glacial CH_3COOH
- communal burette of $\text{CH}_3\text{CH}_2\text{OH}$
- communal burette of deionised H_2O
- communal burette of 1.0 mol dm^{-3} of HCl solution

- 2 x 100 cm^3 conical flask with stopper
- 2 x 250 cm^3 conical flask
- 50 cm^3 burette and funnel
- burette clamp and stand
- 1 cm^3 measuring cylinder (or 1 cm^3 pipette with filler)
- 100 cm^3 measuring cylinder

- Deionised water in wash bottle
- Standard solution of 0.1 mol dm^{-3} sodium hydroxide solution
- Phenolphthalein

Safety considerations

1.0 mol dm^{-3} HCl solution	- irritant
glacial CH_3COOH	- irritant
$\text{CH}_3\text{CH}_2\text{OH}$	- flammable

Method part 1

1. Prepare the control flask and reaction flask by mixing the appropriate chemicals in the quantities listed in the table below:

All volumes in cm^3

		The control	The mixture
Reagent	Density/ g cm^{-3}		
glacial ethanoic acid	1.05	0.0	6.0
ethanol	0.79	0.0	6.0
water	1.00	18.0	6.0
1 mol dm^{-3} hydrochloric acid	1.00	2.0	2.0
Total volume		20.0	20.0

2. Stopper, label and shake both flasks and leave them for at least one week.
3. Using the densities and volumes given for each reagent, calculate the initial amount of CH_3COOH , $\text{CH}_3\text{CH}_2\text{OH}$ and H_2O in moles. You should assume that 2.0 cm^3 of the 1 mol dm^{-3} HCl solution adds an extra 2.0 cm^3 of H_2O to the mixture.

Method part 2

1. Empty the contents of the control flask mixture into a 250 cm^3 conical flask.
2. Add 100 cm^3 of deionised H_2O and 2 drops of phenolphthalein to the flask.
3. Titrate the mixture against the standard solution of NaOH. Record the results of this titration.
4. Transfer 1.0 cm^3 of the reaction mixture to a 250 cm^3 conical flask.
5. Add 100 cm^3 of deionised water and 2 drops of phenolphthalein to the flask.
6. Swirl the flask and titrate the mixture against the standard solution of sodium hydroxide solution as quickly as possible.
7. Repeat steps 4 to 6 with further 1.0 cm^3 samples of the equilibrium mixture until you have three concordant results.
8. Use the results of the titrations to calculate the equilibrium constant of the reaction.

TITRATION USING A pH PROBE

Specification reference: A2 Unit 3.9

Aim

To obtain titration curves for strong and weak acids and alkalis

Apparatus and chemicals

- access to deionised water
- 0.1 mol dm⁻³ HCl solution
- 0.1 mol dm⁻³ CH₃COOH solution
- 0.1 mol dm⁻³ NaOH solution
- 0.1 mol dm⁻³ NH₃ solution

- 25 cm³ bulb/volumetric pipette with safety filler
- 50cm³ burette and funnel
- burette clamp and stand
- 100 cm³ beaker
- 250 cm³ conical flask
- pH meter
- data logger (optional)

- pH 4 buffer solution
- pH 9 buffer solution
- 0.1 mol dm⁻³ HCl solution
- 0.1 mol dm⁻³ CH₃COOH solution
- 0.1 mol dm⁻³ NaOH solution
- 0.1 mol dm⁻³ NH₃ solution

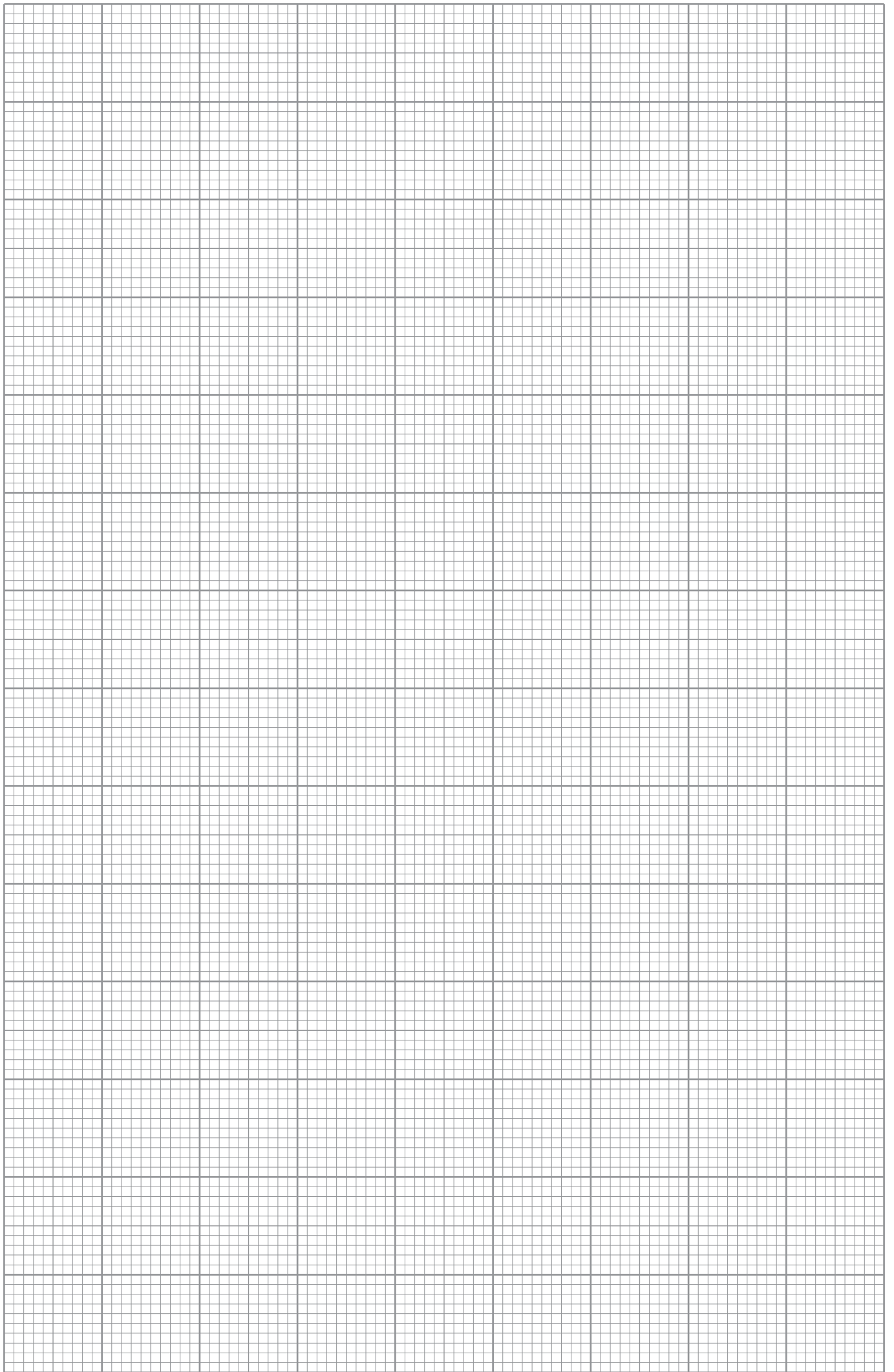
Safety considerations

pH 4 buffer solution	- irritant
pH 9 buffer solution	- irritant
0.1 mol dm ⁻³ HCl solution	- irritant
0.1 mol dm ⁻³ CH ₃ COOH solution	- irritant
0.1 mol dm ⁻³ NaOH solution	- irritant
0.1 mol dm ⁻³⁻³ NH ₃ solution	- irritant



Method

1. Calibrate the pH meter by immersing the electrodes in the pH 4 buffer solution, swirl the beaker, and adjust the reading to 4.0. Remove the electrode, wash with distilled water and immerse in the pH 9 buffer solution. The pH should read 9.0.
2. Using the pipette and filler, transfer 25.0cm^3 of acid to the conical flask and measure the pH.
3. Add the alkali, 5cm^3 at a time up to 50cm^3 , and measure the pH after each 5cm^3 addition. Wash the electrode with distilled water between measurements.
4. If there is time available, repeat the procedure for all combinations of acid and alkali. If not, collaborate with other students so that your class has at least one set of results for each of the following combinations:
 - HCl and NaOH
 - CH_3COOH and NaOH
 - HCl and NH_3
 - CH_3COOH and NH_3
5. Plot graphs of volume of alkali against pH. Read off the pH at the equivalence point for each titration.



SYNTHESIS OF A LIQUID ORGANIC PRODUCT

Specification reference: A2 Unit 4.8

Aim

To synthesise 2-chloro-2-methylpropane using 2-methylpropan-2-ol and concentrated hydrochloric acid and purify the product using a separating funnel followed by distillation

Apparatus and chemicals

- access to deionised water
- access to 2 decimal place mass balance

- 100 cm³ round bottom flask
- 100 cm³ conical flask
- 100 cm³ conical flask with stopper
- 10 cm³ measuring cylinder
- 50 cm³ measuring cylinder
- 50 cm³ separating funnel
- ice bath
- reflux condenser
- thermometer
- sample vial
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand
- filter funnel with cotton wool

- (CH₃)₃COH
- concentrated HCl solution
- granular anhydrous CaCl₂
- saturated NaHCO₃ solution
- anhydrous MgSO₄ powder

Safety considerations

(CH ₃) ₃ COH	- flammable
concentrated HCl solution	- corrosive
CaCl ₂	- irritant



Method

1. Add 30 cm³ of concentrated HCl solution to a stoppered conical flask. This step should be performed in a fume cupboard.
2. Cool the HCl solution in the ice bath.
3. When the HCl solution has cooled add 2.5 g anhydrous CaCl₂ to the solution.
4. Add 10 cm³ of (CH₃)₃COH to the reaction mixture and mix thoroughly.
5. Remove the mixture from the ice bath and allow it to slowly warm up to room temperature. Note that a gaseous side product can be formed at this stage and so the stopper should be released periodically to alleviate any build-up of pressure.
6. Transfer the reaction mixture to the separating funnel.
7. Stopper the funnel and invert it a few times to thoroughly mix the reaction mixture. You may need to alleviate the pressure in the funnel by holding it upside down and opening the tap.
8. Run off and discard the aqueous layer. If you are unsure which layer is the aqueous layer, add deionised water to funnel and see which layer increases in volume.
9. Add 10 cm³ of saturated NaHCO₃ solution to the organic layer and mix thoroughly in the separating funnel. CO₂ gas is formed in this step so it is important to alleviate the pressure in the funnel periodically.
10. Run off and discard the aqueous layer.
11. Repeat steps 9 and 10 to wash the product for the second time.
12. Repeat steps 9 and 10 using 15 cm³ of deionised water to wash the product for a third time.
13. Transfer the organic product to a conical flask, add anhydrous MgSO₄ and swirl vigorously. Keep adding MgSO₄ until it no longer clumps together.
14. Transfer the organic product to the round bottomed flask through a filter funnel with a plug of cotton wool to remove the MgSO₄.
15. Set up the distillation apparatus.
16. Distil off the (CH₃)₃CCl produced and collect in a clean, dry, pre-weighed 100 cm³ conical flask.
17. Record the temperature at which the liquid product is collected.
18. Calculate the mass of the product and use this information to calculate the product yield (Note that the density of (CH₃)₃COH is 0.775 g cm⁻³)

Boiling point of 2-chloro-2-methylpropane	
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SYNTHESIS OF A SOLID ORGANIC PRODUCT

Specification reference: A2 Unit 4.8

Aim

To synthesise 2-acetoxybenzenecarboxylic acid (aspirin) from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride, purify the product using recrystallization and determine its melting point

Apparatus and chemicals

- access to deionised water
- access to 3 decimal place mass balance (minimum 2 decimal place)

- 25 cm³ pear shaped flask
- 10 cm³ measuring cylinder
- hot water bath
- ice bath
- glass stirring rod
- Buchner funnel
- suction apparatus
- watch glass
- sample vial
- labels/suitable pen
- filter paper
- melting point apparatus / Thiele tube
- capillary tube

- 2-hydroxybenzenecarboxylic acid
- (CH₃CO)₂O
- concentrated H₃PO₄ solution

Safety considerations

2-hydroxybenzenecarboxylic acid	- harmful
(CH ₃ CO) ₂ O	- flammable, corrosive
concentrated H ₃ PO ₄ solution	- corrosive

Method

1. Weigh out 1.0 g of 2-hydroxybenzenecarboxylic acid and transfer to a pear shaped flask.
2. Add 2 cm³ of (CH₃CO)₂O and 8 drops of concentrated H₃PO₄ solution.
3. Connect the reflux condenser and place in a fume hood.
4. Warm the mixture in a hot water bath until all of the solid dissolves then warm for a further 5 minutes.
5. Carefully add 5 cm³ of cold deionised water to the solution and stand the flask in a bath of iced water until precipitation appears to be complete. It may be necessary to stir vigorously with a glass rod to start the precipitation process.
6. Filter the mixture under reduced pressure to obtain the impure derivative and wash with a little cold water.
7. Using a boiling tube in a water bath, dissolve the impure product in the minimum amount of warm ethanol.
8. Add 5 cm³ of warm water drop-wise. If the solution becomes cloudy, heat until it becomes clear again.
9. Place the boiling tube into ice water for 15 minutes or until the crystals stop forming.
10. Filter the purified derivative under reduced pressure.
11. Dry the purified product using filter paper.
12. Collect your sample in a dry, pre-weighed sample vial and calculate the mass of the product. Use this information to calculate the percentage yield of the product.
13. Measure the melting point of the product.

Melting point of 2-acetoxybenzenecarboxylic acid	
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TWO-STEP SYNTHESIS

Specification reference: A2 Unit 4.8

Aim

To synthesise 3-nitrobenzenecarboxylic acid via nitration of methyl benzenecarboxylate followed by alkaline hydrolysis of the ester functional group

Apparatus and chemicals

- access to cold deionised water
- access to a 3 decimal place mass balance (minimum 2 decimal place)
- access to ice

- spatula
- weighing boat
- 2 x 100 cm³ conical flask
- 25 cm³ measuring cylinder
- ice bath
- glass Pasteur pipette with rubber teat
- glass stirring rod
- 250 cm³ beaker
- Buchner funnel
- suction apparatus
- filter paper
- 10 cm³ measuring cylinder
- hot water bath
- round bottomed flask
- reflux condenser
- thermometer
- sample vial
- labels/suitable pen
- anti-bumping granules
- heating mantle / Bunsen burner with water bath
- clamp stand
- melting point apparatus / Thiele tube
- capillary tube

- concentrated H₂SO₄ solution
- C₆H₅COOCH₃
- concentrated HNO₃ solution
- CH₃OH
- NaOH
- concentrated HCl solution
- 0.1 mol dm⁻³ HCl solution

Safety considerations

concentrated H ₂ SO ₄ solution	- corrosive
C ₆ H ₅ COOCH ₃	- flammable
concentrated HNO ₃ solution	- corrosive
CH ₃ OH	- flammable, toxic
NaOH	- corrosive
concentrated HCl solution	- corrosive
0.1 mol dm ⁻³ HCl solution	- irritant

Method part 1

1. Carefully add 20 cm³ of concentrated H₂SO₄ solution to a conical flask.
2. Add 10.2 g of C₆H₅COOCH₃ to the conical flask.
3. Cool the reaction mixture to 0 °C in an ice bath.
4. Slowly add, with stirring, 12.5 cm³ of a 1:1 mixture of concentrated H₂SO₄ solution and concentrated HNO₃ solution. Monitor the temperature of the reaction with a thermometer as you perform this step and ensure that the temperature does not rise above 10 °C.
5. Pour the reaction mixture slowly onto ice in a beaker.
6. When the ice has melted, filter the mixture under reduced pressure.
7. Wash the solid once with cold deionised water and twice with cold CH₃OH.
8. Recrystallise the solid product from hot methanol using a hot water bath.
9. Dry the recrystallized product, record its mass and measure its melting point.
10. Use the mass of the product to calculate the percentage yield.

Method part 2

1. To a round bottomed flask add 9.0 g of your product from part 1, 20 cm³ of deionised water and 4.0 g of NaOH.
2. Attach a condenser to the round bottomed flask and heat the reaction mixture under reflux for 5-10 minutes.
3. Carefully pour the reaction mixture, with stirring, into 12.5 cm³ of concentrated HCl solution. This step should be performed in a fume cupboard.
4. Cool the reaction mixture to room temperature using an ice bath.
5. Filter the mixture under reduced pressure and wash with cold deionised water.
6. Recrystallise the solid from hot 0.1 mol dm⁻³ HCl solution using a hot water bath.
7. Dry the recrystallized product, record its mass and measure its melting point.
8. Use the mass of the product to calculate the percentage yield.

Melting point of 3-nitrobenzenecarboxylate	
Melting point of 3-nitrobenzenecarboxylic acid	

PLANNING A SEQUENCE OF TESTS TO IDENTIFY ORGANIC COMPOUNDS

Specification reference: A2 Unit 4.4

Aim

Plan and then carrying out a sequence of tests to identify organic compounds from a given list

Apparatus and chemicals

- access to deionised water
- access to disposable gloves (or suitable long-term use gloves such as nitriles)
- test tubes with stoppers
- boiling tubes
- small beakers
- test tube racks
- red and blue litmus or similar indicator papers
- 2 mol dm⁻³ NaOH solution
- 1 mol dm⁻³ H₂SO₄ solution
- NaHCO₃
- 2,4-DNPH
- materials for preparing Tollens' reagent
- materials for preparing the reagent for the iodoform test
- NaNO₂

Safety considerations

2 mol dm ⁻³ NaOH solution	- irritant
1 mol dm ⁻³ H ₂ SO ₄ solution	- irritant
2,4-DNPH	- flammable, toxic
0.1 mol dm ⁻³ NH ₃ solution	- irritant
0.1 mol dm ⁻³ AgNO ₃ solution	- harmful
NaNO ₂	- oxidising, toxic, dangerous to the environment
CH ₃ CH ₂ CH ₂ CH ₂ NH ₂	- flammable, harmful
C ₆ H ₅ CHO	- flammable, harmful
CH ₃ COCH ₂ CH ₃	- flammable, irritant
C ₆ H ₅ COC ₆ H ₅	- irritant
C ₆ H ₅ COOC ₂ H ₅	- irritant
CH ₃ CONH ₂	- carcinogenic
C ₆ H ₅ CN	- harmful, irritant



General method

1. You are provided with a sample of 6 unknown organic chemicals. These chemicals may be any six from the following eight chemicals.

1-aminobutane	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$
benzenecarbaldehyde	$\text{C}_6\text{H}_5\text{CHO}$
benzenecarboxylic acid	$\text{C}_6\text{H}_5\text{COOH}$
diphenylmethanone	$\text{C}_6\text{H}_5\text{COC}_6\text{H}_5$
butanone	$\text{CH}_3\text{COCH}_2\text{CH}_3$
ethanamide	CH_3CONH_2
ethylbenzenecarboxylate	$\text{C}_6\text{H}_5\text{COOC}_2\text{H}_5$
benzenecarbonitrile	$\text{C}_6\text{H}_5\text{CN}$

2. You are also provided with the apparatus and chemicals listed on the previous page.

Planning

1. Complete a written method detailing how you will identify these chemicals in the fewest possible steps. This method should include a diagram showing the sequence of tests and the outcomes for each sequence.
2. Complete a full risk assessment for this investigation.

PAPER CHROMATOGRAPHY

Specification reference: A2 Unit 4.8

Aim

To analyse biro inks using paper (or thin layer) chromatography

Apparatus and chemicals – paper chromatography

- Whatman Grade 1 chromatography paper
- scissors
- green, black, blue and red biro inks (or equivalent)
- 250 cm³ bottle or conical flask with tight fitting bung
- Pasteur pipette
- developing solvent* (20 cm³ of water, 20 cm³ of ethanol and 60 cm³ butan-1-ol)

Safety considerations – paper chromatography

developing solvent - flammable

Method – paper chromatography

1. Cut a piece of chromatography paper to fit the bottle or conical flask and draw a straight pencil line approximately 10-15 mm from the bottom edge of the paper.
2. Make sure the biro is working and then make a small dot (no more than 1-2 mm in diameter) on the paper on the pencil line. If more than one biro dot (e.g. different colours) is placed on the line, ensure that there is at least a 5 mm gap between the dots along the line. Do not put any dots close to the side edges of the chromatography paper.
3. Add the developing solvent to the bottle to a depth of no more than 10 mm. Use a fume cupboard when using the developing solution.
4. Carefully insert the paper into the solvent. Make sure that the pencil line with the biro dots stays above the solvent level. Hold the chromatography paper in place with a cork or bung.
5. Leave the flask/bottle where it will not be moved for about 30 minutes. Do not allow the solvent front to reach the bottom of the bung.
6. Remove the paper, mark the position of the solvent front and leave it to dry near an open window or in a fume cupboard.
7. The dry chromatograms can be stuck into the lab book.

Note: thin layer chromatography may be used in place of paper chromatography.

