Planning Experiments

[Planning]: Titration 1 – Acid-Base

1 Citrus fruits such as limes, lemons and oranges are an excellent source of vitamins and they also contain a mixture of weak acids such as citric acid, tartartic acid and ascorbic acid.

You are required to plan an experiment involving titration that will enable you to determine an accurate value for the concentration of H^+ present in lemon juice. (Assume that the acid present in lemon juice is a monobasic acid.)
You are provided with:

- **FA 1** 100 cm^3 of fresh (filtered) lemon juice
- **FA 2** 400 cm^3 of 0.150 mol dm^{-3} aqueous sodium hydroxide
- Phenolphthalein and methyl orange indicator
- Deionized water
- Common laboratory apparatus

(a) It is found that 1 drop of **FA 1** required 5 drops of **FA 2** for complete reaction. Explain why the given **FA 1** should not be titrated with **FA 2** directly.

(b) State the dilution factor for the **FA 1** solution so that 25 cm^3 of this diluted solution would require approximately 25 cm^3 of **FA 2** for complete reaction.

(c) Outline, step by step, how you would prepare 250 cm^3 of diluted solution of **FA 1** based on your answer in **part (b)**.

(d) Give a detailed sequence of steps to produce a set of consistent titres that could be used to find the concentration of H^+ present in lemon juice.

In your plan, you should include

- details of apparatus and quantities of reagent to be used;
- the essential details of the titration procedure;
- the appropriate data to be recorded .

(e) A student carried out your plan and obtained an average titre value of **y** cm^3. Outline how you would use this result to determine the concentration of H^+ present in **FA 1**.

[Planning]: Titration 2 – Acid-Base

2(a) Outline a stepwise titration method by which you can determine whether a solid organic acid provided is monobasic or dibasic.

You are provided with:

- 1.0 g of an anhydrous solid organic acid of M_r of 90
- 0.010 mol dm^{-3} aqueous sodium hydroxide
- deionized water
- common laboratory apparatus

In your plan, you should

- indicate the mass of acid to be used and the indicator;
- justify the amount and concentration of the chemicals used and,
- show the appropriate recording of data.

(b) Explain how you would interpret the data to determine obtain the basicity of the organic acid.

[Planning]: Titration 3 – Redox Reaction

3 You are to devise a method to determine the percentage purity of potassium nitrite in an impure sample **Y**. The percentage purity of KNO_2 in the given sample **Y** is around 95% – 98%.

Nitrite ions, NO_2^-, are oxidised to NO_3^- by acidified potassium manganate(VII), $KMnO_4$. However, nitrite ions decompose in contact with dilute acids.

$$NO_2^-(aq) + H^+(aq) \rightarrow HNO_2(aq)$$
$$3HNO_2(aq) \rightarrow H_2O(l) + HNO_3(aq) + 2NO(g)$$

You are provided with:

- solid sample **Y**
- 0.20 mol dm^{-3} potassium manganate(VII)
- 1.0 mol dm^{-3} sulfuric acid

(a) Bulk solution of **Y** is prepared by dissolving 1 g of **Y** in deionised water to form 250 cm^3 of solution. Using calculations, explain why the 0.20 mol dm^{-3} $KMnO_4$ solution provided is **not** suitable for the titration in this case. [Hint: % purity of KNO_2 in the given sample **Y** is around 95% – 98%]

(b) Give an appropriate sequence of steps which enable you to use titration results to determine the percentage purity of KNO_2. Your plan should include

- suitable starting quantities of $KMnO_4$ and H_2SO_4 to carry out the above titration by considering the answer in part **(a)**.
- steps to prepare solution **Y** mentioned in part **(a)**.
- steps taken to prevent significant decomposition of NO_2^- ion taking place during titration. You may assume that the rate of redox reaction is faster than rate of decomposition of NO_2^- ion caused by acid.
- the endpoint colour change by considering the colour of the ions in reactants and products.

(c) Show, step by step, how you will calculate the percentage purity of potassium nitrite in sample **Y**. Assume that the titration result is 22.50 cm^3.

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[Planning]: Titration 4 – Iodine-thiosulfate

4 Iodine is an essential element in the human body. It is normally obtained through the diet but in regions of many countries there is an iodine deficiency.

Sodium chloride, NaC*l*, used in food industry can have a very small amount of potassium iodide, KI, added as a source of iodine. The product is sold as 'iodised salt'.

In the manufacture of 'iodised salt', sodium chloride and potassium iodide are dry mixed to give a mixture containing 2000 parts per million of potassium iodide.

> In a manufacturing of 'iodised salt', too much potassium iodide was accidentally added to the sodium chloride.
>
> You are required to **outline** a plan involving titration to determine the percentage by mass of potassium iodide present in the 'iodised salt' sample.

You are provided with:

- 0.100 mol dm^{-3} copper sulfate
- 0.100 mol dm^{-3} of thiosulfate ions
- starch solution
- 'iodised salt'
- usual apparatus for titration
- an electronic balance

In your plan, you should include

- masses and volumes of reagents to be used from the preliminary consideration **(You may assume 20 g of iodised salt can easily dissolve in deionised water to form 25.0 cm^3 of solution.)**
- reagents that must be in excess
- how the mass of potassium iodide is to be calculated from the titre obtained by working with arbitrary values like **A** cm^3

[Planning]

[Planning] : Gas Collection 1

When the nitrates of the Group 1 metals (Li, Na, K, Rb, Cs) are heated they decompose in one of the following ways.

$$2M\text{NO}_3(s) \rightarrow 2M\text{NO}_2(s) + \text{O}_2(g)$$

$$4M\text{NO}_3(s) \rightarrow 2M_2\text{O}(s) + 4\text{NO}_2(g) + \text{O}_2(g)$$

M represents a Group 1 metal.

You are to design an experiment in which the **oxygen** gas evolved from the thermal decomposition of a known quantity of potassium nitrate is collected and its volume measured.

Nitrogen dioxide, NO_2, is **soluble** in water.

Oxygen gas, O_2, can be regarded as **insoluble** in water.

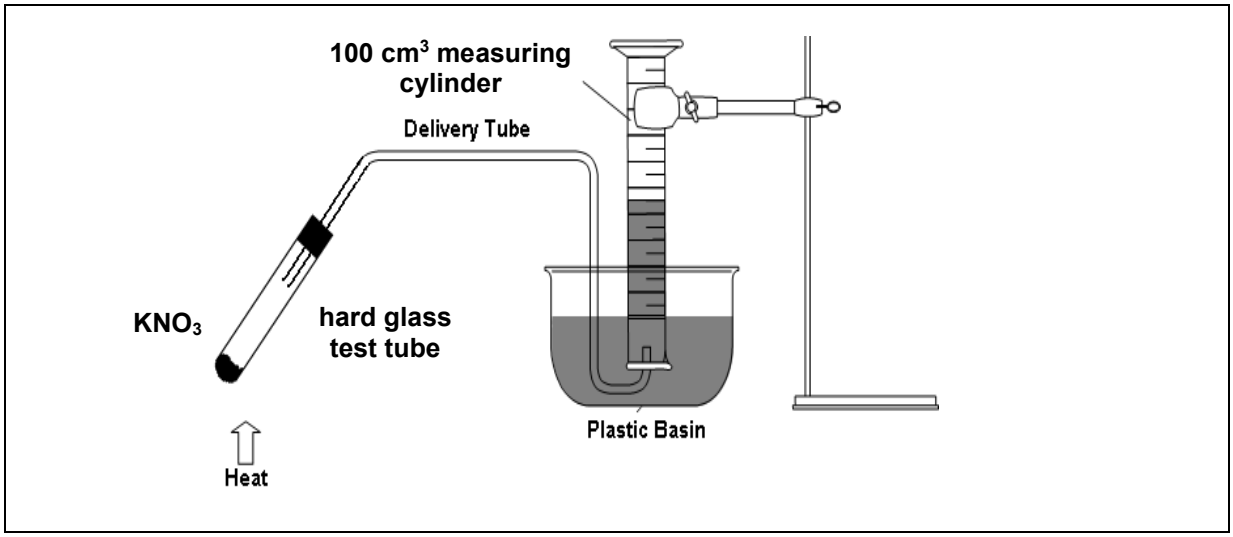
From the data collected in the experiment, you should be able to determine which of the above equations correctly represents the decomposition of potassium nitrate.

(a) Draw a labelled diagram of the apparatus you would use to

- decompose the potassium nitrate by heating,
- remove any nitrogen dioxide formed,
- collect the oxygen gas evolved, and
- measure the volume of the gas collected.

Your diagram should show how standard pieces of laboratory apparatus are to be assembled.

You should show clearly how these pieces of apparatus are connected together.



(b) What is the maximum volume of oxygen gas that can be collected in the apparatus shown in your diagram?

(c) **(i)** For each of the following equations, calculate the mass of potassium nitrate, KNO_3, that, when completely decomposed, will produce the volume of oxygen you have stated in **(b).**

(You may assume that all gases are measured at room temperature and pressure.)

$2KNO_3(s) \rightarrow 2KNO_2(s) + O_2(g)$

$4KNO_3(s) \rightarrow 2K_2O(s) + 4NO_2(g) + O_2(g)$

(ii) What is the maximum mass of potassium nitrate that should be used in the experiment? Explain your answer.

(d) List, in the order in which they will be made, the measurements you would take during the experiment.

(e) The residue after heating the potassium nitrate will be either potassium oxide, K_2O, or potassium nitrite, KNO_2.

Use your knowledge of the chemistry of metal oxides to suggest an appropriate test and observations to distinguish between potassium oxide and potassium nitrite.

[Planning] : Gas Collection 2

GRAVIMETRY, GAS COLLECTION, BACK TITRATION

A solid sample contains a mixture of NaC*l* and $Mg(HO_3)_2$. The mixture contains approximately 30% by mass of magnesium hydrogencarbonate.

The analysis of the solid mixture can be carried out using three methods:

(i) gravimetric analysis
(ii) gas collection
(iii) back titration

You are provided with the following chemicals:

- 0.01 mol dm^{-3} NaOH(aq)
- 1.0 mol dm^{-3} HC*l*
- solid mixture of NaC*l* and $Mg(HCO_3)_2$

Copy the table into your answer sheet and complete it.

	Preliminary calculation to determine suitable starting quantities of the chemical reagents	Outline of step-by-step procedure taking precautions to ensure reliability of results	Table of data to be collected
Gravimetric analysis			
Gas collection			
Back titration			

Gas collection

$Mg(HCO_3)_2 + 2HCl \rightarrow MgCl_2 + 2CO_2 + 2H_2O$

① $V_{gas\ collected}$

Vol of gas collected = 80% of 100 cm^3 gas syringe

= 80 cm^3.

② n_{CO_2}

Amount of $CO_2 = \frac{80}{24000} = 3.333 \times 10^{-3}$ mol

③ $n_{Mg(HCO_3)_2}$

Amount of $Mg(HCO_3)_2$ to produce 80 cm^3 of CO_2

$= \frac{1}{2}(3.333 \times 10^{-3}) = 1.667 \times 10^{-3}$ mol

④ $mass\ Mg(HCO_3)_2$

Mass of $Mg(HCO_3)_2 = 1.667 \times 10^{-3} \times 146.3 = 0.2438$ g

Since the mixture contains approximately 30% by mass of magnesium hydrogencarbonate,

⑤ mass of solid mix.

mass of solid mixture required $= 0.2438 \div \frac{30}{100}$

$= 0.813$g

Given that $[HCl] = 1.0$ mol dm^{-3},

⑥ V_{HCl}

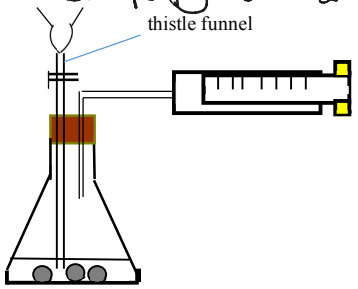
volume of HCl required to react with $Mg(HCO_3)_2$

$= (3.33 \times 10^{-3}) \div \frac{1.0}{1000} = 3.33\ cm^3$

To ensure complete reaction, an excess of HCl of 10 cm^3 is used.

1. Weigh accurately about 0.80 g of solid mixture in a clean and dry weighing bottle using an electronic balance, to be added directly into a 250cm^3 conical flask. Record data in Table 1. ⇒ prep. of reagents.
2. Set up the apparatus according to the diagram below using a 100.0 cm^3 frictionless gas syringe.

↳ specify issue in expt.



↗ take note of precision

3. Add 10.0 cm^3 HCl using a 10.0 cm^3 measuring cylinder into a graduated dropping funnel/thistle funnel. ⇒ measurements
   ↳ note apparatus ↖
4. Open the tap to release 5.0 cm^3 of HCl into the conical flask and leave some HCl behind. Then close the tap. (it's important to use excess HCl so as to achieve air-tight conditions at all times) ⇒ process
   * minimise contamination into flask.
5. Record the volume of the gas collected (to 1 decimal place) when no more effervescence is seen or the piston of the syringe stopped moving (constant value), indicating reaction is complete, wait for a few minutes (cool down) before reading the volume of CO_2 in the gas syringe and record the result. ⇒ observations & recordings

consider what can contribute to readings?

6. (if repeat expt) Repeat steps 1-5 and check that the difference in V_{CO2}/ mass used of two experiments is within 5% difference. ⇒ reliability.

Table 1

	1	2
Mass of weighing bottle and solid mixture/ g		
Mass of weighing bottle and residual solid / g		
Mass of solid transferred / g		
Volume of CO_2/ cm^3		
V_{CO2}/ mass used		

(a) A student carried out gravimetric analysis and back titration to verify the percentage by mass of $Mg(HCO_3)_2$ in the solid mixture.

The following data were obtained

Gravimetric analysis	Back titration
Mass of solid mixture = 1.850 g Mass of residue after repeated heating = 1.453 g	Volume of 0.01 mol dm^{-3} of NaOH(aq) needed to neutralise the unreacted acid in 25.0 cm^3 = 25.60 cm^3 Mass of solid mixture = 0.950 g Total volume of 1 mol dm^{-3} HC*l* used to prepare 250 cm^3 solution = 6.60 cm^3

Use the above data to determine the percentage by mass of $Mg(HCO_3)_2$ in solid mixture for each method used. [29.6%, 31.1%]

Planning Exercises

[Planning]: Gravimetric Analysis Practical 1

1 Sodium carbonate, Na_2CO_3, does not decompose on heating with a Bunsen burner. In contrast, sodium hydrogencarbonate, $NaHCO_3$, decomposes on heating.

$$2\ NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$$

You are to design an experiment in which the percentage by mass of $NaHCO_3$ in a mixture of Na_2CO_3 and $NaHCO_3$ can be determined by heating and weighing alone.

You are provided with:

- solid sample containing a mixture of Na_2CO_3 and $NaHCO_3$
- boiling tube and test-tube holder
- electronic balance
- Bunsen burner and heat proof mat

(a) Your plan should give a step-by-step description of the method, including

- appropriate weighings you would take,
- the sequence of steps to ensure that decomposition was complete,
- the recordings using the letters A, B, C etc to represent the measurement taken and present it with an appropriate table format.

(b) Use the letters from your plan in **(a)** to show how you would process the results to find the percentage by mass of $NaHCO_3$ in the mixture.

(c) The solid sample should be heated gently at first before applying strong heating for thermal decomposition to occur. Suggest a reason why this is necessary.

(d) By considering the products of the decomposition, suggest a reason why a crucible, without a lid, might be more appropriate than a boiling tube.

[Planning]: Gravimetric Analysis Practical 2

2 When aqueous sodium chloride, NaC*l*, is added to aqueous lead nitrate, $Pb(NO_3)_2$, a white precipitate of lead chloride, $PbCl_2$, is produced. A suggested stoichiometric equation is as shown.

$$Pb(NO_3)_2(aq) + 2NaCl(aq) \rightarrow PbCl_2(s) + 2NaNO_3(aq)$$

In separate experiments, different volumes of 0.20 mol dm^{-3} aqueous sodium chloride are added to a fixed volume of 0.10 mol dm^{-3} aqueous lead nitrate. In each case, the precipitate is filtered, washed with deionised water and thoroughly dried. The mass of the precipitate is recorded.

You are to plan an experiment to investigate this reaction in order to confirm or reject the stoichiometry of the equation.

(a) By considering the suggested stoichiometric equation, predict and explain how the number of moles of the precipitate, $PbCl_2$, will change as the number of moles of NaC*l* added increases.

(b) State a limiting factor that must be taken into account when increasing the volume of the aqueous sodium chloride added.

Sketch the graph which would result if, after some of the experiments, the NaC*l* is in excess. Start your graph with no NaC*l* added.

(c) In the experiment you are to plan, identify the following:

(i) independent variable

(ii) dependent variable

(iii) another variable to be controlled

(d) Design a laboratory experiment to test your prediction in **(a)**.

You are provided with 250 cm^3 of 0.20 mol dm^{-3} aqueous sodium chloride.

(i) Outline how you would prepare 250 cm^3 of 0.10 mol dm^{-3} aqueous lead nitrate.
[*A*r: N, 14.0; O, 16.0; Pb, 207]

(ii) Give a step by step description of how you would carry out **one** experiment.
In your plan, you should state:

- the volumes of each solution to be used,
- how the volumes will be measured,
- how you would dry the precipitate.

(e) Prepare a table to:

Show data you would record when carrying out your experiments and the values you would calculate in order to construct a graph to support or reject your prediction in (**a)**.

Planning Experiment

[Planning] : Thermochemistry 1

1 Given:

The enthalpy change of neutralisation of strong acid and strong base is -57.3 kJ mol^{-1}.

The enthalpy change of neutralisation of weak acid and strong base is approximately -54 kJ mol^{-1}.

You are required to plan an experiment using the solutions provided below to determine the enthalpy change of ionisation of ethanoic acid.

$$CH_3CO_2H(aq) \rightarrow CH_3CO_2^-(aq) + H^+(aq)$$

The enthalpy change of ionisation of ethanoic acid cannot be measured directly. Hence, the construction of an energy cycle and applying of Hess' Law are required.

You are provided with:

- dilute ethanoic acid, 2.00 mol dm^{-3}
- dilute sodium hydroxide, 2.00 mol dm^{-3}
- 0.2 ^{o}C division thermometer
- Styroform cups and lid

In your plan, you should include

- details of apparatus and quantities of reagents to be used;
- procedures involved in the experiment;
- table to show appropriate data to be capture and use the letters A, B, C etc to represent the measurement taken;
- how you would construct energy cycle, use the experiment results (with the letters) for calculation of enthalpy change and apply Hess' Law to determine the enthalpy change of ionization of ethanoic acid.

[Planning] : Thermochemistry 2

2 Zinc reacts with aqueous copper(II) sulfate as shown by the following equation

$$Zn(s) + CuSO_4(aq) \rightarrow Cu(s) + ZnSO_4(aq) \quad \Delta H \approx -200 \text{ kJ mol}^{-1}$$

Using an excess of Zn powder, the reaction takes a **few minutes** to go to completion.

Note: zinc, copper and all of their compounds are toxic.

A 1.50 mol dm^{-3} solution of copper(II) sulfate is provided.

Apparatus provided are:

- two 100 cm^3 styrofoam cups and lids
- 0.2 ^oC division thermometer
- 100 cm^3 measuring cylinder
- electronic balance
- weighing bottle
- 250 cm^3 beaker
- stop watch
- burette

Using the information above, describe how you would determine the enthalpy change of the reaction between zinc and copper(II) sulfate.

In your plan, you must include

- the volume of copper(II) sulfate solution to be used;
- a suitable mass of zinc to be used and its justification;
- description of the experiment you would perform with details of the apparatus and the precautions you would take to determine a more accurate enthalpy change of reaction. (**A diagram is not essential but may help your answer**.)
- how you would use the experimental results to calculate the enthalpy change of the reaction between zinc and copper(II) sulfate.
- the potential safety hazards and the relevant safety precautions you would take.

You may find the following questions useful for preliminary calculations.

(i) Suggest and explain a suitable volume of $CuSO_4$ solution to be used for the above reaction.

(ii) Calculate the amount of $CuSO_4$ present in the suggested volume and the expected rise in temperature on reaction with zinc. Hence discuss whether there is a need to make adjustments to the chemical reagents provided.

(iii) With reference to the answer in part **(ii)**, provide details of how the adjustment should be made.

(i) By considering the precision of the apparatus used to measure the mass and temperature, calculate the total percentage uncertainty incurred in these measurements. Suggest how the percentage error can be reduced.

[Planning] : Thermochemistry 3

3 You are to plan an experiment to determine the enthalpy change per mole of magnesium for the reaction shown below:

$$Mg(s) + ZnSO_4(aq) \rightarrow Zn(s) + MgSO_4(aq)$$

You are supplied with

- 100 cm^3 of 0.50 mol dm^{-3} sulfuric acid
- magnesium ribbon
- pieces of zinc metal (granulated zinc)
- the usual laboratory equipment

(a) Explain how you would use the chemical reagents provided to devise an alternative pathway which enables you to calculate a value for the enthalpy change per mole of magnesium for the reaction by applying Hess' law.

$$Mg(s) + ZnSO_4(aq) \rightarrow Zn(s) + MgSO_4(aq)$$

(b) Given the following information, suggest the quantities of reagents you would use to carry out reactions. justify your answer through appropriate calculations or explanation:

- When 0.2 g of Mg reacts with 50 cm^3 of H_2SO_4, it causes a temperature rise of 3.6 ^oC.
- Enthalpy change of reaction between Zn and H_2SO_4 is approximately +80 kJ mol^{-1}.

(c) Give step-by-step description of how the experiments are going to be carried out. Stating clearly

- apparatus used for **measurements**
- suitable data for collection of data
- how the data collected can be used to determine the unknown enthalpy change

(d) Suggest three modifications that you might make to the experiment to obtain a more accurate result. Justify your answers.

(e) The experiments that you have planned in **(a)** and **(b)** are to be repeated using the same volumes of 0.50 mol dm^{-3} hydrochloric acid in place of the sulfuric acid.

Explain what differences, if any, it would make to the results.

[Planning] : Thermochemistry 4

4 You are provided with solutions **FA 3, FA 4** and **FA 5.** The solutions are:

- 1.0 mol dm^{-3} sodium hydroxide
- 1.0 mol dm^{-3} sulfuric acid
- 0.5 mol dm^{-3} sulfuric acid

You are to plan experiments that will enable you to identify the solution that matches each of **FA 3**, **FA 4** and **FA 5**.

You have the following apparatus available:

- –10 °C to 110 °C thermometer
- 100 cm^3 beaker

a measuring cylinder (Note: No indicator is provided)

(a) You are to identify, by the **minimum number** of practical steps, the solution which contains sodium hydroxide.
Outline your method – with an explanation of the expected results.

Your experiment(s) must use only the solutions and apparatus provided above.

(b) Plan further experiments to find which of the remaining solutions is 1 mol dm^{-3} sulfuric acid and 0.5 mol dm^{-3} sulfuric acid respectively.

Outline your method – with an explanation of the expected results.

Planning

[Planning] : Kinetics Practical 1

Iodide ions, I^-, reacts with peroxodisulfate ions, $S_2O_8^{2-}$ as shown:

$$S_2O_8^{2-}(aq) + 2I^-(aq) \rightarrow 2SO_4^{2-}(aq) + I_2(aq) \text{.....(1)}$$

Colourless blue black with starch

The rate of this reaction maybe followed by measuring the time taken to reach an identifiable point early in the reaction.

An identifiable point of reaction (1) is the appearance of blue black iodine-starch complex.

A small and fixed amount of thiosulfate is used to fix the amount of iodine produced by reaction (1) in every repeated experiment. This is because blue-black colouration of an iodine-starch complex appears only when thiosulfate ion, $S_2O_3^{2-}$, present in the mixture have completely reacted with the iodine as shown in equation (2).

$$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq) \text{ .....(2)}$$

colourless

You are to plan an experiment to investigate how the rate of reaction between $K_2S_2O_8$ and KI depends on the concentration of $S_2O_8^{2-}$.

(a) A preliminary experiment, using approximate volumes of solution, indicates that time taken for the blue-black colouration to appear doubles when $K_2S_2O_8$ (aq) is diluted with an equal volume of water.

Using the results of the preliminary experiment, deduce the order of reaction with respect to $S_2O_8^{2-}$.

(b) Identify **(i)** the independent variable and **(ii)** dependent variable in the experiment described in part **(a).**

(i) independent variable:

(ii) dependent variable:

(c) **(i)** Explain why it is important for the iodine formed via oxidation in reaction (1) is reduced back to iodide ions by thiosulfate.

(ii) Hence explain why the volume of sodium thiosulfate should be measured precisely, using a burette, in each of the experiments.

(d) You are provided with

- 0.60 mol dm^{-3} potassium iodide, KI
- 0.20 mol dm^{-3} potassium peroxodisulfate, $K_2S_2O_8$
- 0.01 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$
- deionised water
- starch indicator
- the equipment normally found in laboratory

A first experiment is carried out using the following quantities

Chemical reagent	KI	$K_2S_2O_8$	$Na_2S_2O_3$	starch solution
Volume used / cm^3	20	60	5	5

By considering the points raised in part **(a)** - **(c)**, give a step-by-step description of how you would carry out **5** further experiments to verify that the order of reaction with respect to $S_2O_3^{2-}$ is 1.

In your plan, you are to include:

- the apparatus you would use
- the procedure you would follow
- the measurements you would make
- a table that records the dependent and independent variables.

(e) Suggest how the results of the experiment can be manipulated in three different ways to verify the answer to part **(d)**.

[Planning] : Kinetics Practical 2

The reaction between ethanedioate ions and acidified manganate(VII) ions as shown below takes place very slowly in the cold but can be accelerated with heat or with addition of Mn^{2+} catalyst.

$$2MnO_4^- + 5C_2O_4^{2-} + 10H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$$

The reaction rate for the reaction can be measured by noting the time elapsed for the purple colour of the manganate(VII) ions to disappear.

(a) A teacher demonstrates this experiment as follows:

She prepares two separate solutions **A** and **B**.

- Solution **A** contains 200 cm^3 of 0.08 mol dm^{-3} ethanedioic acid and 200 cm^3 of 0.50 mol dm^{-3} sulfuric acid
- Solution **B** contains a mixture of 400 cm^3 of 0.0080 mol dm^{-3} potassium manganate(VII) solution and 400 cm^3 of 0.0080 mol dm^{-3} manganese(II) sulfate solution.

(b) She mixes 20 cm^3 of solution **A** with 60 cm^3 of solution **B**. The time taken for the purple manganate(VII) solution to decolourise is about 40 s.

By considering the information given above, you are to plan experiments to study the effects of different concentration of catalyst, $MnSO_4$, on rate of reaction between the ethanedioate ions and acidified manganate(VII) ions.

The order of the reaction with respect to $MnSO_4$ may be determined by performing a number of experiments and then analysis by plotting a suitable graph.

Write a plan to show how the series of experiments can be carried out. In your plan, you should use the same proportions of **A** and aqueous $KMnO_4$ as described above.

You should ensure that at least one of your experiments would be expected to take significantly less than 40 sec.

You may assume that you are provided with the following:

- Solution **A**, having the concentrations of reagent given above
- 0.008 mol dm^{-3} potassium manganate(VII) solution
- 0.08 mol dm^{-3} manganese(II) sulfate solution
- Deionised water
- White tile
- The apparatus normally found in a school or college laboratory

Your plan should contain the following:

- the intended concentration of the stock solution of manganese(II) sulfate solution to be used
- details for the preparation of this stock solution from the manganese(II) sulfate solution provided
- appropriate quantities and concentrations of solutions

- all essential experimental details
- an outline of how the results would be used to determine the order with respect to manganese(II) sulfate.

[Planning] : Kinetics Practical 3

Catalysed decomposition of aqueous hydrogen peroxide gives O_2 is as shown:

$$2H_2O_2\,(aq) \rightarrow 2H_2O(l) + O_2(g)$$

You are provided with:

- 0.10 mol dm^{-3} aq H_2O_2
- 100 cm^3 gas syringe
- Catalyst $Fe(OH)_3$
- The equipment normally found in a school laboratory

(a) Give a step-by-step description of how the volume of oxygen produced is measured over time to monitor the rate of decomposition of H_2O_2.

Your plan should include:

- Given that the measurements are carried out under room conditions and pressure, calculate a suitable volume of H_2O_2 (aq) which gives a total volume of O_2 gas hat occupies 80% of the gas syringe.
- a labelled diagram of the experimental set-up, showing the apparatus you would use
- the procedure you would follow
- the measurements you would take
- how the data measured enable you to determine the order with respect to H_2O_2.

(b) Identify another variable in the experiment that can also affect the volume of O_2 collected. Hence suggest an improvement that can make the experimental results collected more reliable.

[Planning] : Kinetics Practical 4

The decomposition of hydrogen peroxide can be catalysed by Fe(III) chloride solution

$$2H_2O_2 \rightarrow 2\ H_2O + O_2$$

The rate equation for this reaction is: rate = $k[H_2O_2][Fe^{3+}]$ where k is the rate constant.

The order with respect to H_2O_2 maybe confirmed to be first order by preparing a reaction mixture containing H_2O_2 (aq) and $FeCl_3$(aq).

Portions of the reaction mixture are

- Removed at timed intervals,
- Quenched by adding to ice water
- Titrated against standard solution of $KMnO_4$(aq).

The volume of $KMnO_4$ added in each titration (titre) is proportional to the $[H_2O_2]$.

$$2MnO_4^- + 6\ H^+ + 5H_2O_2 \rightarrow 2Mn^{2+} + 5O_2 + 8H_2O$$

(a) Describe how you would make a 100 cm^3 of acidified $KMnO_4$ of concentration 0.05 mol dm^{-3} from $KMnO_4$ of original concentration 0.1 mol dm^{-3} and H_2SO_4 (aq). Label this solution as **FA 1**. You are going to use **FA 1** in part **(b)**.

(b) Plan a procedure to collect sufficient data to allow a graph of *volume of $KMnO_4$ against time to be drawn* to verify that the order of reaction with respect to H_2O_2 is 1.

You should plan to make a reaction mixture containing,

- 100 cm^3 H_2O_2(aq)
- 10 cm^3 of $FeCl_3$(aq)

You are provided with:

FA 1 prepared by you	$FeCl_3$(aq)
aq H_2O_2	ice water

normal apparatus found in the laboratory

In your plan, you are to include:

- the apparatus you would use
- the procedure you would follow
- the measurements you would make

(c) In the space below, sketch the graph you would expect to obtain**.**
Explain your answer.

[Planning] : Inorganic Qualitative Analysis Practical 1

1. **FA2** is a green solution containing the following cations: $Al^{3+}(aq)$, $Fe^{3+}(aq)$ and $Cu^{2+}(aq)$.

(a) How does the colour of **FA2** indicate that both the transition metal cations quoted are present?

(b) State, with reasons, whether each of the following anions could also be present.

i. $NO_3^-(aq)$
ii. $SO_4^{2-}(aq)$
iii. $CO_3^{2-}(aq)$

(c) State the reagent, or reagents, needed to test for the presence of $SO_4^{2-}(aq)$.

(d) Plan a procedure to separate the three cations $Al^{3+}(aq)$, $Fe^{3+}(aq)$ and $Cu^{2+}(aq)$ in **FA 2** so that each cation is present in separate precipitates.

You may assume that you are provided with:

- aqueous sodium hydroxide, NaOH,
- aqueous nitric acid, HNO_3,
- aqueous ammonia, NH_3,
- aqueous sodium carbonate, Na_2CO_3,
- dilute hydrochloric acid, HCl,
- deionised water,
- the equipment normally found in a school or college laboratory.

In your plan you should:

- **only** be using the chemicals provided, but **not** necessarily all of them,
- outline the sequence of steps you would follow (You may present using a flow chart),
- give brief details of the quantities of chemicals that you would use i.e. in excess,
- state the observation you would observe,
- identify the cations by the formulae of the precipitates obtained after each separation step.

[Planning] : Inorganic Qualitative Analysis Practical 2

2. There are three aqueous solutions. Each of the solutions contains one of the following:

   - magnesium sulfate
   - sodium hydroxide
   - zinc nitrate

   Plan a series of test-tube experiments which will enable you to identify the compounds present in each aqueous solution using only the three solutions and aqueous barium chloride. Use of any form of indicator or additional reagent will be is not allowed.

   You may assume that you are provided with:
   - three unknown aqueous solutions,
   - aqueous barium chloride,
   - the equipment normally found in a school or college laboratory.

   In your plan you should:

   - **only** be using the chemicals provided,
   - outline the sequence of steps you would follow (You may present using a flow chart),
   - highlight the chemicals that you would use in excess,
   - state the observation you would observe,
   - identify the solution by the formulae of the precipitates obtained after each separation step.

[Planning] : Inorganic Qualitative Analysis Practical 3

3. In this question, you are to plan an experiment to identify the five colourless solutions listed below.

    - ammonium sulfate,
    - barium chloride,
    - dilute hydrochloric acid,
    - sodium sulfate,
    - sodium sulfite.

    The five solutions are in unlabelled bottles.

    Plan a series of test-tube tests which will enable you to identify the compound present in each bottle.

    You may assume that you are provided with:

    - five unknown aqueous solutions,
    - aqueous sodium hydroxide,
    - red and blue litmus paper,
    - the equipment normally found in a school or college laboratory.

    In your plan you should:

    - **only** be using the chemicals provided,
    - outline the sequence of steps you would follow (You may present using a flow chart),
    - highlight the chemicals that you would use in excess,
    - state the observation you would observe,
    - state the positive test you would conduct to identify the gas evolved (if any),
    - provide brief explanations of how you identify the solution in each bottle.

[Planning] : Inorganic Qualitative Analysis Practical 4

**The $E^{\ominus}$ concept will be taught in the elctrochemistry topic

4. **FA3** is a dilute aqueous solution which is believed to contain one of the cations, barium or magnesium, and a halide anion (chloride, bromide or iodide).

   You are required to devise an experiment which will enable you to identify the ions present in **FA 3**. You may use **ONLY** the reagents provided below.

aqueous barium chloride	dilute nitric acid
aqueous sodium hydroxide	dilute sulfuric acid
aqueous hydrogen peroxide (an oxidant)	dilute hydrochloric acid
aluminium foil	dilute aqueous ammonia

   Half equations

$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O$	$E^{\ominus} = + 1.77$ V
$Cl_2 + 2e^- \rightarrow 2Cl^-$	$E^{\ominus} = + 1.36$ V
$Br_2 + 2e^- \rightarrow 2Br^-$	$E^{\ominus} = + 1.07$ V
$I_2 + 2e^- \rightarrow 2I^-$	$E^{\ominus} = + 0.54$ V

(a) Plan two test tube experiments to distinguish between barium and magnesium ions in **FA 3**.

In your plan you should:
- **only** be using the chemicals provided,
- an outline of the sequence of steps you would follow,
- highlight the chemicals that you would use in excess,
- state the observation you would observe,
- identify the cations by the formulae of the precipitates obtained in each test.

(b) Plan one test tube experiment to identify the anion present in **FA 3**.

In your plan you should:
- **only** be using the chemicals provided,
- outline the step you would follow,
- state the observation you would observe,
- how you identify the anion based on the observation.

[Planning] : Organic synthesis Practical 1

1. The preparation of *N-phenylethanamide* from phenylamine is termed ***acylation.*** Both ethanoyl chloride, CH_3COCl, and ethanoic anhydride, $(CH_3CO)_2O$, may be used as acylating agents. The reactivity of ethanoic anhydride is lower than that of ethanol chloride, and allows the reaction rate to be more easily controlled. Phenylamine is most conveniently used in the form of the salt phenyl ammonium chloride.

The ***acylation*** reaction is performed in two stages.

Stage 1: A solution of sodium ethanoate is treated with phenylammonium chloride, forming phenylamine, ethanoic acid and sodium chloride.

Stage 2: Phenylamine then reacts with ethanoic anhydride to form *N-phenylethanamide* as a white solid, together with ethanoic acid.

An aqueous solution of phenylammonium chloride (5.0 g in 150 cm^3) is mixed with ethanoic anhydride (10 cm^3). To this mixture is added a second aqueous solution of sodium ethanoate (30 g in 125 cm^3). The sodium ethanoate causes the reaction in step 1 to occur. Once phenylamine is released, it reacts readily with ethanoic anhydride to form *N*- phenylethanamide as a white solid. The crude solid product is isolated and recrystallised from water.

Note: An excess of ethanoic anhydride is used to ensure a good yield.

(a) Write an equation for each of the two stages in the preparation of *N-phenylethanamide*.

Stage 1:

Stage 2:

(b) Assuming that this reaction typically yields around 70% of the theoretical maximum, calculate the mass of phenylammonium chloride needed to make 2.0 g of *N-phenylethanamide*.

(c) Write a plan to obtain a **pure** and **dry** crystalline *N-phenylethanamide* from the crude solid N-phenylethanamide obtained from stage 2.

You may assume that you are provided with:

- the equipment normally found in a school or college laboratory.

In your plan, you should include details of:

- the apparatus you would use,
- the procedure you would follow to obtain pure and dry crystalline N-phenylethanamide.

(d) Describe how you will assess the purity of the product obtained and how to interpret the results.

(e) Suggest a reason why a student may obtain a higher yield than expected.

(a) Identify a potential safety hazard and suggest a safety precaution to minimise this risk.

[Planning] : Organic Qualitative Analysis Practical 2

2. 1–iodopropane can be synthesized from 1–bromopropane and sodium iodide, using dry propanone as a solvent.

$$CH_3CH_2CH_2Br + NaI \rightleftharpoons CH_3CH_2CH_2I + NaBr$$

(a) **Table 1** shows the solubilities of sodium bromide and sodium iodide in propanone.

Table 1

compound	solubility at 55 °C in g / 100 g of propanone
sodium bromide	0.00841
sodium iodide	39.9

Use this information in **Table 1**, explain why the reaction produces a very high yield of $CH_3CH_2CH_2I$.

(b) The preparation of 1–iodopropane is described as follows:

1–bromopropane, excess sodium iodide and propanone solvent are mixed and then heated under reflux conditions for around thirty minutes.

The crude product is then shaken with water, before the layer containing 1–iodopropane is separated out and distilled to isolate pure 1–iodopropane.

Table 2 gives the boiling points and densities of 1–bromopropane, 1–iodopropane and propanone.

Table 2

compound	boiling point / ^oC	density / g cm^{-3}
1–bromopropane	71.0	1.35
1–iodopropane	102.6	1.75
propanone	56.0	0.784

[A_r: C, 12.0; H, 1.0; O, 16.0; Br, 79.9; I, 126.9; Na, 23.0]

(i) Assuming that the reaction goes to completion, calculate the volume of 1–bromopropane and mass of sodium iodide you would need to prepare **at least 10.0 g** of pure 1–iodopropane. Show your working clearly.

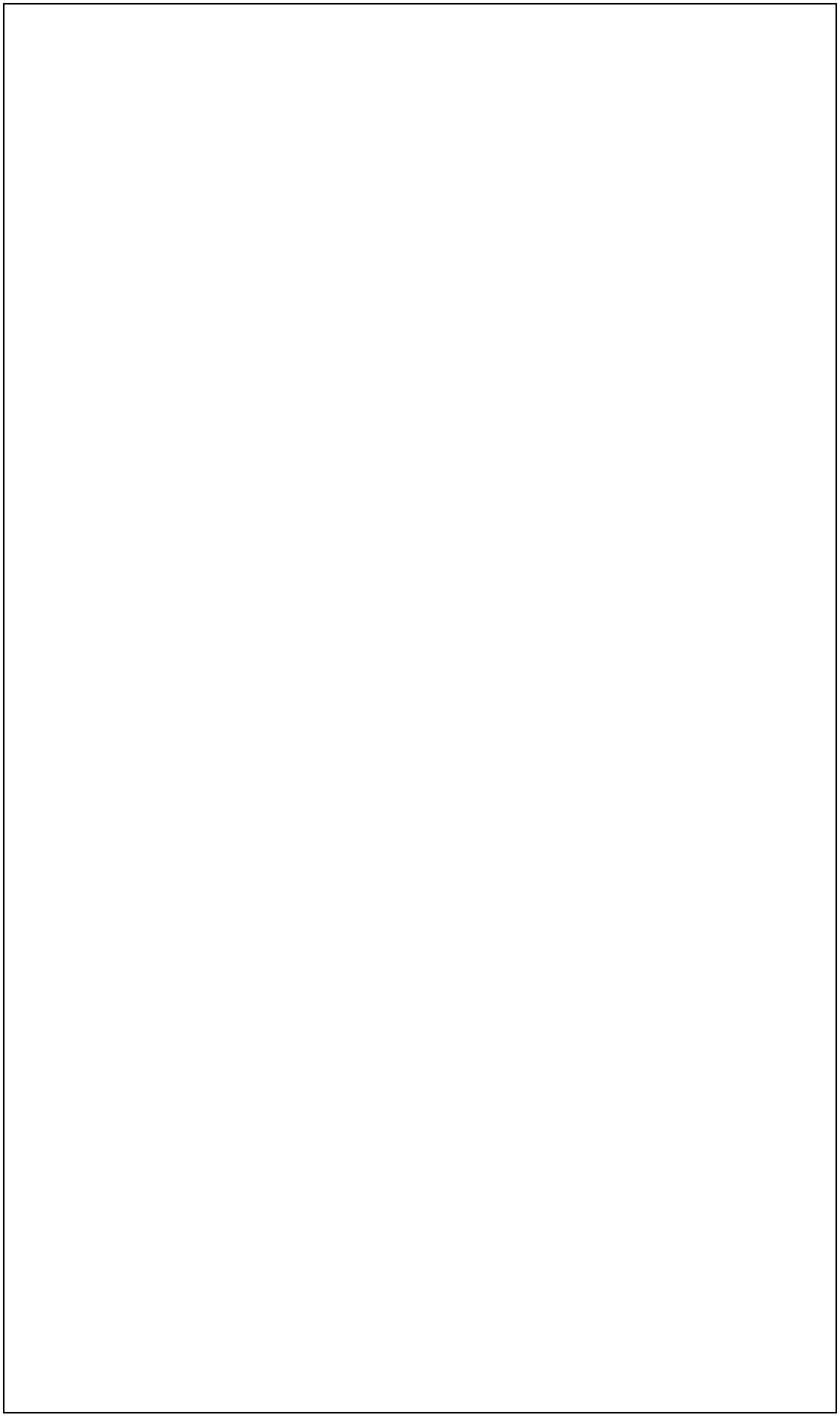
(ii) Write a plan for the preparation of **at least 10.0 g** of pure and dry 1–iodopropane.

You may assume that you are provided with:

- liquid 1-bromopropane,
- solid sodium iodide,
- 25.0 cm^3 liquid propanone,
- the equipment normally found in a school or college laboratory.

In your plan, you should include details of:

- the quantities of the reactants calculated in **(b)(i)**,
- the apparatus you would use, including well-labelled diagrams of the assembled apparatus,
- the procedure you would follow to obtain pure and dry 1–iodopropane.



[Planning] : Organic Qualitative Analysis Practical 3 (2024 A level P4)

Consider the following six organic compounds, which are all liquids at room temperature.

C butanal	**D** butan-1-ol	**E** butan-2-ol
F 2-methylpropan-2-ol	**G** pentan-2-one	**H** pentan-3-one

Plan an investigation, using test-tube reactions, which would allow you to identify each of the these six organic compounds.

Your plan should include:

- Details of the reagents to be used
- An outline of the sequence of steps you would follow
- How the results of your tests would identify each compound.

Compounds may be identified by positive or negative results in the tests.

You should use as few different tests as possible.

You may present your answer in a flow-chart or table. [8]