Data Processing Exercises

[Data Processing]: Volumetric Analysis Practical 1

1. Determination of the number of molecules of H_2O of crystallisation in borax

In this experiment, you are to determine the number of moles of H_2O in hydrated borax.

Borax, also known as disodium tetraborate, reacts with hydrochloric acid according to the equation bxelow:

$$Na_2B_4O_7\,(aq) + 2HCl\,(aq) + 5H_2O\,(l) \rightarrow 2NaCl\,(aq) + 4H_3BO_3\,(aq)$$

You are given the following:

FA 1 is 109.30 g dm^{-3} borax, $Na_2B_4O_7.xH_2O$
FA 2 is 0.100 mol dm^{-3} HCl
Indicator

Procedure

1. Using a burette, place between 17.80 cm^3 and 18.20 cm^3 of **FA 1** into a 100 cm^3 volumetric flask. Record your burette reading below.
2. Make up to the mark with deionised water and shake thoroughly. Label this solution **FA 3**.
3. Fill in a second burette with **FA 2**.
4. Pipette 25.0 cm^3 of **FA 3** into a conical flask and add a few drops of indicator provided.
5. Titrate the contents of the conical flask with **FA 2** until the end point indicated by a colour change.
6. Repeat the titration as many times as necessary to obtain consistent results.
7. Tabulate your results in an appropriate manner.

Results

Table 1: Dilution of FA 1

Final burette reading / cm^3	18.00
Initial burette reading / cm^3	0.00
Volume of **FA 1** used / cm^3	18.00

Table 2: Titration of FA 3 against FA 2

	Rough	1	2
Final burette reading / cm^3	25.95	25.80	26.40
Initial burette reading / cm^3	0.00	0.00	0.60
Volume of **FA 2** used / cm^3	25.95	25.80	25.80
Consistent results		√	√

(a) **(i)** Using your titration results, calculate the number of moles of the $Na_2B_4O_7$ in 25.0 cm^3 of **FA 3**.

no. of moles of HCl in 25.80 cm^3 = $\frac{25.80}{1000}$ x 0.100 = 2.58 x 10^{-3} mol

no. of moles of $Na_2B_4O_7$ in 25.0 cm^3 = ½ (2.58 x 10^{-3}) = 1.29 x 10^{-3} mol

number of moles of $Na_2B_4O_7$ in 25.0 cm^3 of **FA 3** = 1.29 x 10^{-3} mol [1]

(ii) Calculate the number of moles of $Na_2B_4O_7$ in 100 cm^3 of **FA 3**.

no. of moles of $Na_2B_4O_7$ in 100 cm^3 = 1.29 x 10^{-3} x 4 = 5.16 x 10^{-3} mol

number of moles of $Na_2B_4O_7$ in 100 cm^3 of **FA 3** = 5.16 x 10^{-3} mol [1]

(iii) Hence calculate the concentration of $Na_2B_4O_7$ in **FA 1** in mol dm^{-3}.

No. of moles of $Na_2B_4O_7$ in 100 cm^3 of FA 3 = No. of moles of $Na_2B_4O_7$ in 18.00 cm^3 of FA 1 = 5.16 x 10^{-3}

concentration = 5.16 x 10^{-3} / (18.00/1000) = 0.2866 = 0.287 mol dm^{-3}

concentration of $Na_2B_4O_7$ in **FA 1** = 0.287 mol dm^{-3} [1]

(iv) Determine the relative molecular mass of $Na_2B_4O_7.\mathbf{x}H_2O$.

no. of moles of $Na_2B_4O_7.\mathbf{x}H_2O$ in 1 dm^3 = no. of moles of $Na_2B_4O_7$ in 1 dm^3
M_r = 109.30 / 0.2866 = 381.4

M_r of $Na_2B_4O_7.\mathbf{x}H_2O$ = 381.4

Hence calculate the value of **x** in $Na_2B_4O_7.\mathbf{x}H_2O$. Give your answer to the nearest whole number.
[*A*r: H, 1.0; B, 10.8; O, 16.0; Na, 23.0]

x = (381.4 – 23.0 x 2 – 4 x 10.8 – 16.0 x 7) / 18.0
= 10

x is 10 [2]

(b) **(i)** Calculate the oxidation state of boron in $Na_2B_4O_7$ and in H_3BO_3.

oxidation state of B in $Na_2B_4O_7$ is +3 ..... [1]

oxidation state of B in H_3BO_3 is +3 ..... [1]

(ii) Hence suggest the type of reaction between $Na_2B_4O_7$ and HC*l*.

acid base reaction / neutralisation
.....
[1]

(iii) Hence or otherwise, suggest a identity for the indicator used in this titration.

methyl orange / phenolphthalein / thymolphthalein
.....
[1]

(c) Student **A** repeated the experiment, however in step **1**, he transferred **FA 1** to a 250 cm^3 graduated flask by mistake and top up with deionised water to the mark accordingly. He then obtained the titration results as shown below.

	1	2	3
Final burette reading / cm^3	10.00	20.80	30.80
Initial burette reading / cm^3	0.10	10.50	20.40
volume of **FA 2** / cm^3	9.90	10.30	10.40

(i) With reference to his titration results, explain how the mistake affected the reliability of his results.

The **FA 3** solution prepared become 2.5 times more dilute, hence the titre values become 2.5 times smaller. This decreases the reliability of his results as percentage error due to measurement is higher with a smaller titre volume.

(ii) Identify any anomalous titre value in the student's result. Suggest an error that could have lead to the anomalous result.

Experiment 1 as it is not within ±0.10 cm^3 of experiment 2 and 3.

The student did not rinse the pipette with **FA 3** after rinsing with deionised water, the residual water in the pipette diluted the aliquot transferred to the conical flask. Hence lesser amount of $Na_2B_4O_7$ is used, lowering the amount of HC*l* needed to neutralise it, resulting in a smaller titre volume.

2. To calculate the values of a and b in the formula of the ammonium salt $(NH_4)_aFe(SO_4)_b.6H_2O$

Iron(II) ammonium sulfate, $(NH_4)_aFe(SO_4)_b.6H_2O$ dissociates into iron(II) sulfate and ammonium ions when in solution. In this experiment, you will perform a redox titration of $(NH_4)_aFe(SO_4)_b.6H_2O$ with potassium manganate(VII).

You are given the following:

FA 1 is iron(II) ammonium sulfate crystals
FA 3 is 0.0100 mol dm^{-3} potassium manganate (VII)
sulfuric acid solution

You are to record all the mass measurements and titration results in the space provided.

Preparation of FA 2 by dissolving FA 1 in sulfuric acid

1. Weigh accurately about 5.00 g of **FA 1**. Record your readings in an appropriate manner.
2. Using a 50 cm^3 measuring cylinder, transfer 30.0 cm^3 of sulfuric acid to dissolve the crystals in a small beaker.
3. Transfer the solution and washings into a 250 cm^3 graduated flask and make up to the mark with deionised water. Shake the solution thoroughly and label this **FA 2**.

Titration with potassium manganate(VII) solution

4. Pipette 25.0 cm^3 of **FA 2** into a conical flask.
5. Titrate the mixture against **FA 3** until the solution changes from yellow to permanent orange.
6. Repeat the titration as many times as necessary to obtain consistent results.

(a) Results

Table 1: Mass readings

Mass of weighing bottle and iron(II) ammonium salt /g	9.256
Mass of weighing bottle and residual iron(II) ammonium salt /g	4.253
Mass of iron(II) ammonium salt /g	5.003

Table 2: Titration with FA 3

	Rough	1	2
Final burette reading / cm^3	26.50	27.50	25.50
Initial burette reading / cm^3	0.00	2.00	0.00
Volume of $KMnO_4$ used /cm^3	26.50	25.50	25.50
Consistent results		√	√

(b) **(i)** Give the balanced chemical equation for the reaction taking place during titration.

$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

…….. [1]

(ii) Using your titration results, calculate the amount of $KMnO_4$ used in the reaction.

average titre volume = $\frac{25.50+25.50}{2}$ = 25.50 cm^3

no. of moles of $KMnO_4$ used = $\frac{25.50}{1000}$ x 0.01 = 2.55 x 10^{-4} mol

amount of $KMnO_4$ = 2.55 x 10^{-4} mol [1]

(iii) Calculate the amount of Fe^{2+} in 25.0 cm^3 of **FA 2**. Hence, calculate the amount of Fe^{2+} in the mass of **FA 1** used.

No. of moles of Fe^{2+} in 25.0 cm^3 = 5 x 2.55 x 10^{-4} = 1.275 x 10^{-3} mol

No. of moles of Fe^{2+} in 250 cm^3 = $\frac{250}{25}$ x 1.275 x 10^{-3} = 1.28 x 10^{-2} mol

amount of Fe^{2+} = 1.28 x 10^{-2} mol [2]

(c) Upon futher analysis, the amount of NH_4^+ ion in **FA 2** is found to be 2.55 x 10^{-2} mol. Using your answer to **(b)(iii)**, calculate the values of **a** and **b** in the formula $(NH_4)_aFe(SO_4)_b.6H_2O$.

$nNH_4^+ / nFe^{2+} = a$

$2.55 \times 10^{-2} / 1.275 \times 10^{-2} = a = 2$

Looking at charges:
$(NH_4)_2Fe(SO_4)_b$
$2(+1) + (+2) + b(-2) = 0$
$\therefore b = 2$

a is 2 and **b** is 2 [2]

(d) **(i)** Explain the purpose of using sulfuric acid, instead of deionised water, to dissolve **FA 1** in step **2**.

This is to provide an acidic medium for the redox reaction to occur as shown in the chemical equation.

(ii) A student claims that the amount of NH_4^+ in **FA 2** can be determined by directly titrating 25.0 cm^3 of **FA 2** with a known concentration of NaOH. Explain whether you agree with the student's claim.

Disagree, as Fe^{2+} in **FA 2** will form a precipitate with OH^- and the end-point cannnot be observed clearly.

(ii) Using knowledge of qualitative analysis, suggest how you can

I. verify that NH_4^+ and Fe^{2+} ions are present in the **FA 2**.

NH_4^+: Add NaOH(aq), warm the solution and test with moist red litmus paper. Colourless and pungent NH_3 is evolved, turning the moist red litmus paper blue.

Fe^{2+}: Add NaOH(aq), a green precipitate of $Fe(OH)_2$ will be observed.

II. hence separate the NH_4^+ and Fe^{2+} ions in **FA 2** in the form of solution.

Add **excess** NaOH (aq) and filter the resultant mixture.

To the residue, add excess nitric acid to get back Fe^{2+} solution.

To the filtrate, add in excess nitric acid to get back NH_4^+ ions.

3. Determination of % of mass of H_2O in $CuSO_4.nH_2O$

Copper(II) ions reacts with an excess of iodide ions to form a mixture containing white copper(I) iodide precipitate in brown I_2 solution:

Equation 1: $4I^-(aq) + 2Cu^{2+}(aq) \rightarrow I_2(aq) + 2CuI(s)$

The mass of water of crystallisation present in the hydrated copper(II) salt can be calculated by titrating the iodine liberated with sodium thiosulfate.

Equation 2: $2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(s)$

You are provided with

FA 1 is 200 g dm^{-3} hydrated copper(II) sulfate solution
FA 3 is potassium iodide solution
FA 4 is 0.100 mol dm^{-3} sodium thiosulfate solution
Starch indicator

Procedure

1. Using a burette, place exactly 25.00 cm^3 of **FA 1** into a 250 cm^3 volumetric flask. Record your readings in a table.
2. Make up to the mark with distilled water and shake thoroughly. Label this solution **FA 2**.
3. Pipette 25.0 cm^3 of **FA 2** into a conical flask and add about 15.0 cm^3 of **FA 3** using a 50 cm^3 measuring cylinder. This will produce the aforementioned mixture.
4. Titrate the iodine liberated with **FA 4** until the solution turns pale yellow.
5. Add about 1 cm^3 of starch indicator, using a dropper, and continue titration until the blue-black colour is discharged.
6. Repeat the titration as many times as necessary to obtain consistent results.
7. Tabulate your results in an appropriate manner.

Results

Table 1: Dilution of FA 1

Final burette reading / cm^3	25.00
Initial burette reading / cm^3	0.00
Volume of **FA 1** used / cm^3	25.00

Table 2: Titration with FA 4

	Rough	1	2	3
Final burette reading / cm^3	23.55	43.60	20.25	20.30
Initial burette reading / cm^3	0.00	23.55	0.00	0.20
Volume of **FA 4** used / cm^3	23.55	20.05	20.25	20.10
Consistent results		√		√

(a) Calculation

(i) From your titrations, obtain a suitable volume of **FA 4** to be used in your calculations. Show clearly how you obtained this volume.

average volume of $Na_2S_2O_3$ = (20.05 + 20.10) / 2 = 20.08 cm^3

Volume of **FA 4** = 20.08 cm^3 ..... [1]

(ii) Calculate the number of moles of $Na_2S_2O_3$ required to react with the iodine liberated by 25.0 cm^3 of **FA 2**.

number of moles of $Na_2S_2O_3$ = 20.08/1000 x 0.100 = 2.008 x 10^{-3} mol
= 2.01 x 10^{-3} mol

number of moles of $Na_2S_2O_3$ = 2.01 x 10^{-3} mol ..... [1]

(iii) Hence, calculate how many mole of Cu^{2+} were there in 25.0 cm^3 of **FA 2**.

number of moles of Cu^{2+} = number of moles of $S_2O_3^{2-}$
= 2.008 x 10^{-3} mol
= 2.01 x 10^{-3} mol

number of moles of Cu^{2+} = 2.01 x 10^{-3} mol ..... [1]

(iv) Using your answer in **(iii)**, calculate the number of moles of Cu^{2+} in 250 cm^3 of **FA 2**. Hence, calculate the concentration of $CuSO_4.\mathbf{n}H_2O$ in mol dm^{-3} in **FA 1**.

number of moles of $CuSO_4.\mathbf{n}H_2O$ = 2.008 x 10^{-3} x 10 = 2.008 x 10^{-2}

concentration of $CuSO_4.\mathbf{n}H_2O$ = 2.008 x 10^{-2}/ (25/1000)
= 0.8032 mol dm^{-3}
= 0.803 mol dm^{-3}

concentration of $CuSO_4.\mathbf{n}H_2O$ = 0.803 mol dm^{-3} ..... [1]

(v) Determine the relative molecular mass of $CuSO_4.\mathbf{n}H_2O$.

molar mass = $\frac{200}{0.8032}$ = 249.0 g mol^{-1}

M_r = 249.0

M_r of $CuSO_4.\mathbf{n}H_2O$ = 249.0 .....

Hence calculate the value of **n** in $CuSO_4.\mathbf{n}H_2O$. Give your answer to the nearest whole number.
[*A*r: H, 1.0; O, 16.0; S, 32.1; Cu, 63.5]

n = [249.0 – (63.5 + 32.1 + 16.0 x 4)] / 18.0 = 4.97 ≈ 5

n is 5 ..... [2]

(vi) Calculate the percentage mass of water of crystallisation in the salt.

% mass = (5x18.0) / 249.6 = 36.1 %

percentage mass of water of crystallisation 36.1 %.....[1]

(b) Calculate the maximum percentage error in the volume of **FA 4** in your first accurate titre.

% error = (± 0.05 x 2) / 20.05 x 100 = ±0.499 %

percentage error = ±0.499 % ..... [1]

(d) A student carried out this practical and calculated a value of 2 for **n**. The textbook value of **n** is more than 2.

Suggest an error the student could have done in the practical procedure of the experiment that could account for this result and explain why this gives a value of **n** that is too low.

To get a lower value of **n**, the student will get a higher titre value than actual. The student might have rinsed the conical flask with **FA 2** before titration. This results in a larger amount of Cu^{2+} than in 25.0 cm^3 of **FA 2**, thus resulting in a larger titre value. The same mass divided by larger amount of Cu^{2+} will result in a smaller *M*r and hence a smaller value of **n**.

4. Determination of change in oxidation number of unknown element X

In this experiment, you are to determine the change in oxidation number of element **X**, when $\mathbf{X}O_3^-$ ions react with Fe^{2+} ions.

There are two reactions taking place.

$\mathbf{X}O_3^-$ is first reacted with excess Fe^{2+} to give the reduced form of **X** and Fe^{3+}. The unreacted Fe^{2+} is subsequently titrated with acidified $KMnO_4$. The reduced form of **X** does not react with $KMnO_4$.

You are given the following:

FA 1: 0.75 mol dm^{-3} acidified Fe^{2+} ions
FA 2: 0.075 mol dm^{-3} $\mathbf{X}O_3^-$
FA 3: 0.020 mol dm^{-3} $KMnO_4$

Procedure

1. Using a burette, place exactly 50.00 cm^3 of **FA 1** into a 250 cm^3 volumetric flask.
2. Make up to the mark with distilled water and shake thoroughly. Label this solution **FA 4**.
3. Pipette 25.0 cm^3 of **FA 4** into a conical flask.
4. Using the other pipette, add 10.0 cm^3 of **FA 2** to the same conical flask.
5. Titrate the mixture with **FA 3**.
6. Repeat the titration as many times as necessary to obtain consistent results.
7. Tabulate your results in an appropriate manner.

Results

	Rough	1	2
Final burette reading / cm^3	22.65	22.45	44.95
Initial burette reading / cm^3	0.00	0.00	22.45
Volume of **FA 3** used /cm^3	22.65	22.45	22.50
		√	√

(a) (i) Write the balanced chemical equation for the reaction taking place during the titration.

$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

…….. [1]

(ii) Using your titration results, calculate the amount of Fe^{2+} that reacted with MnO_4^-.

average titre volume = $\frac{22.45+22.50}{2}$ = 22.48 cm^3

no. of moles of MnO_4^- in 22.48 cm^3 = $\frac{22.48}{1000}$ x 0.0200 = 4.496 x 10^{-4} mol

no. of moles of Fe^{2+} that reacted with MnO_4^- = 5 x 4.496 x 10^{-4}
= 2.25 x 10^{-3} mol

2.25 x 10^{-3} mol

amount of Fe^{2+} = ... [1]

(iii) Calculate the amount of Fe^{2+} in 25.0 cm^3 of **FA 4** before **FA 2** is added.

concentration of **FA 4** = 0.75 x $\frac{50}{250}$ = 0.15 mol dm^{-3}

amount of Fe^{2+} = ($\frac{25}{1000}$ x 0.15) = 3.75 x 10^{-3} mol

3.75 x 10^{-3} mol

amount of Fe^{2+} = ... [1]

(iv) Hence calculate the amount of Fe^{2+} that reacted with 10.0 cm^3 of **FA 2**.

no. of moles of Fe^{2+} that reacted with **X**O_3^- ions = 3.75 x 10^{-3} – 2.248 x 10^{-3}
= 1.50 x 10^{-3} mol

1.50 x 10^{-3} mol

amount of Fe^{2+} that reacted with 10.0 cm^3 of **FA 2**
= ..... [1]

(v) Calculate how many number of moles of Fe^{2+} ions reacted with one mole of **X**O_3^- ions.

no of moles of **X**O_3^- ions reacted = $\frac{10.0}{1000}$ x 0.075 = 0.00075 mol
no of moles of Fe^{2+} that reacted with one mole of **X**O_3^-
= 1.502 x 10^{-3} / 0.00075
= 2

2

number of moles of Fe^{2+} ions = ... [1]

(vi) Hence, deduce the change in oxidation number of the element **X** when $\mathbf{X}O_3^-$ ion reacts with Fe^{2+} ions.

Oxidation number of **X** in $\mathbf{X}O_3^-$ ion = +5

Since each Fe^{2+} loses 1 electron to form Fe^{3+}, each mole of $\mathbf{X}O_3^-$ reacts with 2 moles of Fe^{2+}, each $\mathbf{X}O_3^-$ gains 2 electrons, hence oxidation number changes from +5 to +3.

[1]

(b) Using your answer to **(a)(vi)**, construct a half-equation for the reaction undergoes by $\mathbf{X}O_3^-$ to form $\mathbf{X}O_2^-$.

Hence give a balanced chemical equation for the reaction between Fe^{2+} and $\mathbf{X}O_3^-$.

$$\mathbf{X}O_3^- + 2H^+ + 2e \rightarrow \mathbf{X}O_2^- + H_2O$$

half equation ..... [1]

$$\mathbf{X}O_3^- + 2H^+ + 2Fe^{2+} \rightarrow \mathbf{X}O_2^- + H_2O + 2Fe^{3+}$$

chemical equation ..... [1]

(c) Explain the importance of performing step **1** and **2** in the procedure, by stating the effect on the titration results if it is not done.

If it is not done, the amount of Fe^{2+} in 25.0 cm^3 is about 5 times greater, which increases the titre by about 5 times and this exceeds the maximum capacity of burette, which makes it unsuitable for titration. Thus, step **1** and **2** is to lower the concentration of Fe^{2+} such that a suitable titre volume will be obtained for titration.

5. Determination of concentration of HC*l* and mass of NaOH in solid mixture through double–indicator titration

When a solution containing a mixture of sodium hydroxide and potassium carbonate is titrated against hydrochloric acid, three reactions occur.

1. $NaOH + HCl \rightarrow NaCl + H_2O$ indicator: *thymolphthalein*
2. $K_2CO_3 + HCl \rightarrow KHCO_3 + KCl$ indicator: *thymolphthalein*
3. $KHCO_3 + HCl \rightarrow KCl + CO_2 + H_2O$ indicator: *methyl orange*

If a solution of this mixture is titrated against hydrochloric acid using thymolphthalein indicator, the blue colour of the indicator is discharged when reactions **1** and **2** are complete.

If methyl orange indicator is then added at this point to the mixture and the titration is continued, the end-point corresponds to the completion of reaction **3**.

You are given the following:

FA 1 is prepared by dissolving a known mass of solid sodium hydroxide with 2.20 g solid potassium carbonate in 250 cm^3 of deionised water in a beaker.
FA 2 is hydrochloric acid of unknown concentration

Titration of FA 1 against FA 2

1. Fill the burette with **FA 2**.
2. Using a 50 cm^3 measuring cylinder, transfer 25.0 cm^3 of **FA 1** into a conical flask and titrate with **FA 2**, using thymolphthalein as indicator.
3. Run **FA 2** from the burette into this flask until solution changes from blue to colourless. Record the volume of acid required.
4. Add 1-2 drops of methyl orange indicator into the reaction mixture in the conical flask and continue titrating until the colour changed from yellow to orange. Record the volume of acid required.
5. Record your titration results in the spaces provided below. Make certain that your recorded results show the precision of your instrument used.
6. Repeat steps **2** to **6** until your results are consistent.

Results

Table 1: Titration of FA 1 with FA 2

	1	2
Initial burette reading / cm^3	0.00	0.00
Burette reading at thymolphthalein end-point / cm^3	21.30	21.20
Burette reading at methyl orange end-point / cm^3	29.30	29.30

(a) Calculation

(i) From your titrations, obtain the average volume of **FA 2** at first end-point and second end-point.

average volume of **FA 2** at first end-point = (21.30 + 21.20) / 2 = 21.25 cm^3

average volume of **FA 2** at second end-point = (29.30 + 29.30) / 2 = 29.30 cm^3

average volume of **FA 4** at first end-point = 21.25 cm^3 [1]

average volume of **FA 4** at second end-point = 29.30 cm^3 [1]

(ii) Calculate the concentration of K_2CO_3 in **FA 1** in mol dm^{-3}.
[*Ar*: C, 12.0; O, 16.0; K, 39.1]

concentration = (2.20/138.2) / (250/1000) = 0.0637 mol dm^{-3}

concentration of K_2CO_3 = 0.0637 mol dm^{-3} [1]

(iii) Using your answer in **(ii)**, calculate the amount of HC*l* required to react with $KHCO_3$ in reaction **3**.

amount of K_2CO_3 used = amount of $KHCO_3$ used = amount of HC*l* needed for reaction **3** = 25/1000 x 0.06367 = 1.59 x 10^{-3} mol

amount of HC*l* required = 1.59 x 10^{-3} mol [1]

(iv) Hence using your titration results and answer in **(iii)**, calculate the concentration of HC*l* in **FA 2**.

volume of HC*l* used for reaction **3** = 29.30 – 21.25 = 8.05 cm^3

concentration of HC*l* used for reaction **3** = 1.591 x 10^{-3} / (8.05/1000) = 0.198 mol dm^{-3}

concentration of HC*l* in **FA 2** = 0.198 mol dm^{-3} [1]

(v) Hence, calculate the mass of NaOH used in the solid mixture to prepare **FA 1**.
[*Ar*: H, 1.0; O, 16.0; Na, 23.0]

From (iii), volume of HC*l* used for reaction with K_2CO_3 in reaction **2** = 8.05 cm^3

volume of HC*l* used to neutralise NaOH = 21.25 – 8.05 = 13.20 cm^3

amount of NaOH in 25 cm^3 = amount of HC*l* for reaction **1** = 13.20 / 1000 x (0.1977) = 2.61 x 10^{-3} mol

mass of NaOH used to prepare 250 cm^3 = 2.61 x 10^{-3} x 10 x 40.0 = 1.04 g

mass of NaOH = 1.04 g …… [2]

(b) **(i)** State one **significant** source of error in the procedure used in this experiment and explain the effect on the titration results. Suggest an improvement that could be made to reduce this error.

One significant error is the use of a measuring cylinder to measure 25 cm^3 of **FA 1** for titration. Measuring cylinder is less precise, which results in a high percentage error due to measurement and hence titration results will be less accurate. A more precise instrument such as pipette should be used to measure **FA 1** instead.

(ii) A student states that the titration results is inaccurate as solid sodium hydroxide, used in preparation of **FA 1**, tends to absorb CO_2 and moisture from the air readily. Explain whether this error will cause the titration results to be higher or lower than expected.

For the same mass of solid used, the actual mass of pure NaOH used is lower and the concentration of NaOH prepared in **FA 1** will be lower than actual. Less HC*l* is required, hence the titre value will be lower than expected.

[Date Processing] : Volumetric Analysis Practical 6

6 Determination of the value of *n* in IO_n^-

IO_n^- is an oxoanion of iodine. It can react with iodide ions, I^-, in an acidic medium, to produce only molecular iodine and water.

This amount of iodine can be analysed via titration with sodium thiosulfate, $Na_2S_2O_3$.

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-$$

In this question, you will perform an experiment to determine the stoichiometric ratio between IO_n^- and I_2 and the value of *n* in IO_n^-.

FA 1 is solid hydrated sodium thiosulfate, $Na_2S_2O_3 \bullet 5H_2O$
FA 2 is a solution containing 0.00825 mol dm^{-3} KIO_n
FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4
FA 4 is 0.25 mol dm^{-3} potassium iodide, KI

You are also provided with starch solution.

(a) Preparation of a standard solution of 0.0484 mol dm^{-3} sodium thiosulfate

(i) Determine the mass of **FA 1** required to prepare 250 cm^3 of sodium thiosulfate solution with a concentration 0.0484 mol dm^{-3}.
[A_r: S, 32.1; O, 16.0; Na, 23.0; H, 1.0]

Amount of $Na_2S_2O_3 = \frac{0.0484}{1000} \times 250 = 0.0121$ mol
M_r of $Na_2S_2O_3 \bullet 5H_2O = 248.2$
Mass of $Na_2S_2O_3 \bullet 5H_2O = 0.0121 \times 248.2 = 3.003 = 3.00$ g

mass of **FA 1** required = …….. g [1]

You will follow the instructions to perform the preparation of the standard solution.

Record your measurements in Table 1.1.

(ii)
1. Using a mass balance, weigh out accurately the mass of **FA 1** determined in **(a)(i)** into a weighing bottle.
2. Dissolve **FA 1** in about 50 cm^3 of deionised water in a 100 cm^3 beaker.
3. Transfer the solution and washings into a 250 cm^3 volumetric flask, using a filter funnel.
4. Top up to the 250 cm^3 mark with deionised water.
5. Stopper the flask and shake to ensure solution is homogeneous. Label this solution as **FA 5**.

Complete Table 1.1 with your mass measurements and calculate the mass of **FA 1** that you used.

Table 1.1

Mass of weighing bottle and **FA 1** / g	
Mass of empty weighing bottle / g	4.020
Mass of weighing bottle and residual **FA 1** / g	
Mass of **FA 1** / g	3.00

[2]

(b) Preparation and titration of the reaction mixture

1. Fill a burette with **FA 5**.
2. Using a 25.0 cm^3 pipette, add 25 cm^3 of **FA 2** to a 250 cm^3 conical flask.
3. Using a measuring cylinder, add 10.0 cm^3 of **FA 3**, followed by 10.0 cm^3 of **FA 4** to the same conical flask and swirl quickly.
4. **Immediately** titrate the I_2 in the conical flask with **FA 5**. When a pale yellow colour is obtained, add 1 cm^3 of starch solution. The end-point is reached when the blue – black colour of the solution decolourises.
5. Repeat steps **2** to **4** until consistent results are obtained and record your results below.

Results

Final burette reading/cm^3	**24.35**	**23.65**
Initial burette reading/cm^3	**0.70**	**0.00**
Volume of FA5/ cm^3	**23.65**	**23.65**

[5]

Obtain, from your titration results, a suitable average titre of **FA 5**. Show clearly the titres you used in calculating this average.

Average volume of **FA5** = (23.65 + 23.65)/ 2 = 23.65 cm^3

Average volume of **FA 5** = …….. [1]

Calculations

Show your working and appropriate significant figures in all of your calculations.

(c) (i) Calculate the amount, in moles, of $Na_2S_2O_3$ present in the volume of **FA 5** determined in **(b)** and hence the amount of iodine produced.

Amount of $S_2O_3^{2-}$ = $0.0484 \times \frac{23.65}{1000}$ = 1.144 x 10^{-3} mol

Amount of I_2 = 0.001144 ÷ 2 = 5.72 x 10^{-4} mol

Amount of I_2 = ……..[2]

(ii) Calculate the amount of **FA 2** used in the titration.

Amount of **FA 2** = $\frac{0.00825}{1000} \times 25 = 2.0625 \times 10^{-4}$
$= 2.06 \times 10^{-4}$ mol

Amount of **FA 2** = ... [1]

(d) **(i)** Construct a balanced half-equation, in terms of *n*, for the reduction of IO_n^- to I_2.

$IO_n^- + (2n-1)e^- + 2nH^+ \longrightarrow \frac{1}{2} I_2 + nH_2O$

..... [1]

(ii) Hence construct a balanced equation, in terms of *n*, for the reaction between IO_n^- and I^- to give I_2 and H_2O.

$IO_n^- + (2n-1)I^- + 2nH^+ \longrightarrow nI_2 + nH_2O$

..... [1]

(iii) Determine the amount of I_2 produced by one mole of IO_n^- and determine the value of *n* in IO_n^-.

Amount of I_2 produced per mole of IO_n^- = $n = \frac{5.72 \times 10-4}{2.06 \times 10-4} = 2.777 \approx 3$

n =..... [1]

7. Determination of K_{sp} of $M(OH)_2$

$M(OH)_2$ is an ionic compound that has a low solubility in water. The solubility of such a compound can be represented using a type of equilibrium constant called the *solubility product, K_{sp}*.

You will determine the solubility product of $M(OH)_2$ by a titration method using sulfamic acid and the indicator screened methyl orange.

You are given the following:

FA 1 is a **saturated** solution of $M(OH)_2$ in water, it is the filtrate formed by adding excess $M(OH)_2(s)$ to a fixed volume of water followed by filtration.
FA 2 is 0.500 mol dm^{-3} sulfamic acid, NH_2SO_3H, a strong monobasic acid
Screened methyl orange

Procedure

1. Using a burette, place between 30.00 and 31.00 cm^3 of **FA 2** into a 250 cm^3 graduated flask and make up to the mark with distilled water. Shake the mixture and label it **FA 3**. Record your burette readings.
2. Pipette 25.0 cm^3 of **FA 1** into a conical flask. Add a few drops of screened methyl orange and titrate with **FA 3**, until it turns from green to grey.
3. Repeat the titration as many times as necessary.
4. Tabulate your results in an appropriate manner.

Results

Table 1: Dilution of FA 2

Final burette reading / cm^3	30.00
Initial burette reading / cm^3	0.00
Volume of **FA 2** used /cm^3	30.00

Table 2: Titration

	rough	1	2
Final burette reading / cm^3	20.50	40.80	20.20
Initial burette reading / cm^3	0.00	20.50	0.00
Volume of **FA 3** used /cm^3	20.50	20.30	20.20
		√	√

(a) (i) Calculate the concentration, in mol dm^{-3}, of sulfamic acid in **FA 3.**

Amount of **FA 2** in 30.00 cm^3 = 30.00 x 10^{-3} x 0.500
= 0.0150 mol

Conc. of sulfamic acid in **FA 3** = 0.01500 / $\frac{250}{1000}$ = 0.0600 mol dm^{-3}

0.0600 mol dm^{-3}

concentration, in mol dm^{-3}, of sulfamic acid in **FA 3** = …… [1]

(ii) Calculate the amount, in moles, of NH_2SO_3H in your mean titre.

average titre volume = $\frac{20.30+20.20}{2}$ = 20.25 cm^3

amount of NH_2SO_3H = 20.25 x 10^{-3} x 0.0600 = 1.22 x 10^{-3} mol

1.22 x 10^{-3} mol

amount of NH_2SO_3H = ... [1]

(b) Give the balanced equation for the neutralisation reaction between $M(OH)_2$ and sulfamic acid in the titration.

$M(OH)_2 + 2NH_2SO_3H \rightarrow M(NH_2SO_3)_2 + 2H_2O$

..... [1]

(c) Calculate the concentration in mol dm^{-3}, of $M(OH)_2$ **FA 1**.

Amount of $M(OH)_2$ in 25.0 cm^3 = ½ amount of NH_2SO_3H
= ½ x 1.215 x 10^{-3}
= 6.075 x 10^{-4} mol

Conc. of $M(OH)_2$ = 6.075 x 10^{-4} / $\frac{25.00}{1000}$ = 0.0243 mol dm^{-3}

0.0243 mol dm^{-3}

concentration, in mol dm^{-3}, of $M(OH)_2$ **FA 1** = ... [1]

(d) Aqueous $M(OH)_2$ is fully ionised in solution **FA 1**.

(i) Use your answer to **(c)** to deduce the concentration of M^{2+} in **FA 1**.

$M(OH)_2$ (aq) → M^{2+} (aq) + $2OH^-$ (aq)
conc. of M^{2+} = conc. of $M(OH)_2$ = 0.0243 mol dm^{-3}

0.0243 mol dm^{-3}

concentration of M^{2+} = ... [1]

(ii) Deduce the concentration of OH^- in **FA 1.**

conc. of OH^- = 2 x conc. of $M(OH)_2$ = 2 x 0.0243 = 0.0486 mol dm^{-3}

0.0486 mol dm^{-3}

concentration of OH^- = ... [1]

(e) **(i)** Give the equation for the solubility product K_{sp}, of $M(OH)_2$, indicating the units.

$K_{sp} = [M^{2+}] [OH^-]^2$ units: $mol^3\ dm^{-9}$

…… [1]

(ii) Use your equation and your answers to **(d)(i)** and **(ii)** to calculate the solubility product of $M(OH)_2$.

$K_{sp} = [M^{2+}] [OH^-]^2$

$= (0.02430) (0.04860)^2$

$= 5.74 \times 10^{-5}\ mol^3\ dm^{-9}$

$5.74 \times 10^{-5}\ mol^3\ dm^{-9}$

K_{sp} of $M(OH)_2$ = …… [1]

(f) Another student performs this experiment using a 50 cm^3 measuring cylinder to measure 30.0 cm^3 of **FA 2** into the graduated flask instead of a burette. State and explain in terms of percentage error (uncertainty), whether this student was wise to choose this measuring cylinder to measure **FA 2.**

% error using measuring cylinder = $\frac{\pm 0.5}{30}$ x 100% = $\pm$1.67%

% error using burette = $\frac{\pm 0.05 \times 2}{30}$ x 100% = $\pm$0.333%

The student is unwise as a larger error due to measurement occurs with the use of the measuring cylinder.

Data Processing

[Data Processing] : *Gas Collection Practical 1*

1. Determination of M_r of the ethanedioate salt

Calcium ethanedioate is the major component of the most common type of human kidney stones. It is one of a series of salts formed from ethanedioic acid, $H_2C_2O_4$. Another of these salts can be represented by the formula $X_2C_2O_4$, where **X** is a Group 1 metal.

Solution **Q** contains 43.0 g dm^{-3} of $X_2C_2O_4$ in deionised water. You are not provided with **Q**.

You are provided with the following chemicals.

FA 1 is a diluted solution of **Q**, in which 35.70 cm^3 of **Q** was made up to 250 cm^3 with deionised water in a graduated flask.
FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.
FA 4 is a solution containing manganese(II) ions, Mn^{2+}.

In this experiment, you will react the ethanedioate salt with potassium manganate(VII), in the presence of a small amount of manganese(II) ions. You will measure the volume of CO_2 gas produced at timed intervals and determine the maximum volume of CO_2 gas produced.

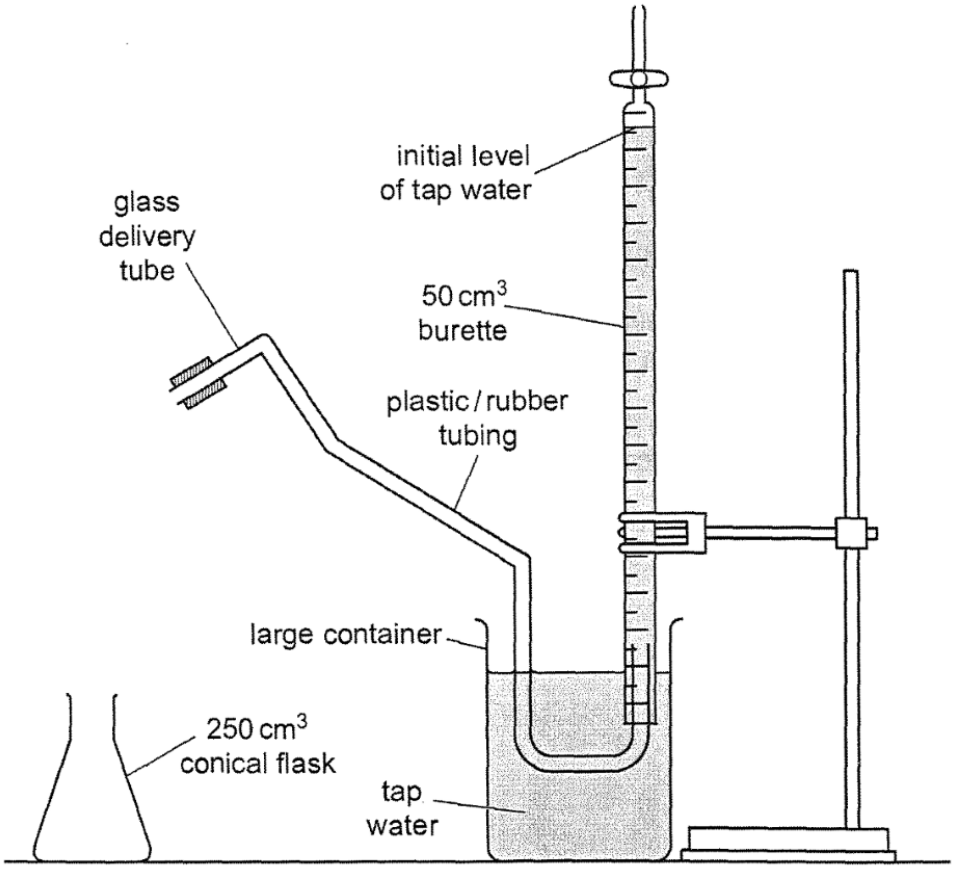


Fig. 1

You will measure the level of water in the burette at timed intervals.

In an appropriate format in the space provided on the next page, prepare a table in which you may record each burette reading and the time it was taken.

In addition, you will need to show the total volume of CO_2 collected up to that time, recorded to one decimal place.

1. Set up the apparatus as shown in Fig 1. You should insert the plastic/rubber tubing to a sufficient depth in the burette so that it will not subsequently shake loose.

2. Adjust the water level in the burette until it is between 48.0 cm^3 and 50.0 cm^3. You may find it helpful to use an empty dropping pipette to introduce small amounts of air from the bottom of the burette tube.

3. Using an appropriate measuring cylinders to add to the 250 cm^3 conical flask
   - 20.0 cm^3 of **FA 1**,
   - 50.0 cm^3 of **FA 3**.

4. Using a dropping pipette, add about 1 cm^3 of **FA 4** to the conical flask.

5. Using an appropriate measuring cylinder, measure out 30.0 cm^3 of **FA 2**.

6. Transfer the **FA 2** into the conical flask and insert the bung into the conical flask.

7. Allow any bubbles created in the burette when the bung was inserted in the conical flask to rise to the top. Start the stopwatch, read and record the initial water level in the burette.

   Note: Once you have started the stopwatch, allow it to continue running for the duration of the experiment. You **must not** stop the stopwatch until the reaction is complete.

8. Check that the plastic/rubber tubing is securely positioned in the burette.

9. Hold the flask by its neck and gently swirl it continuously.

10. At t = 0.5 min, read and record the water level in the burette, to 1 decimal place, together with the time it was measured in your table.

11. Continue to gently swirl the flask. Read and record the water level in the burette every minute, until the reaction is complete.

(a) (i) Experimental results

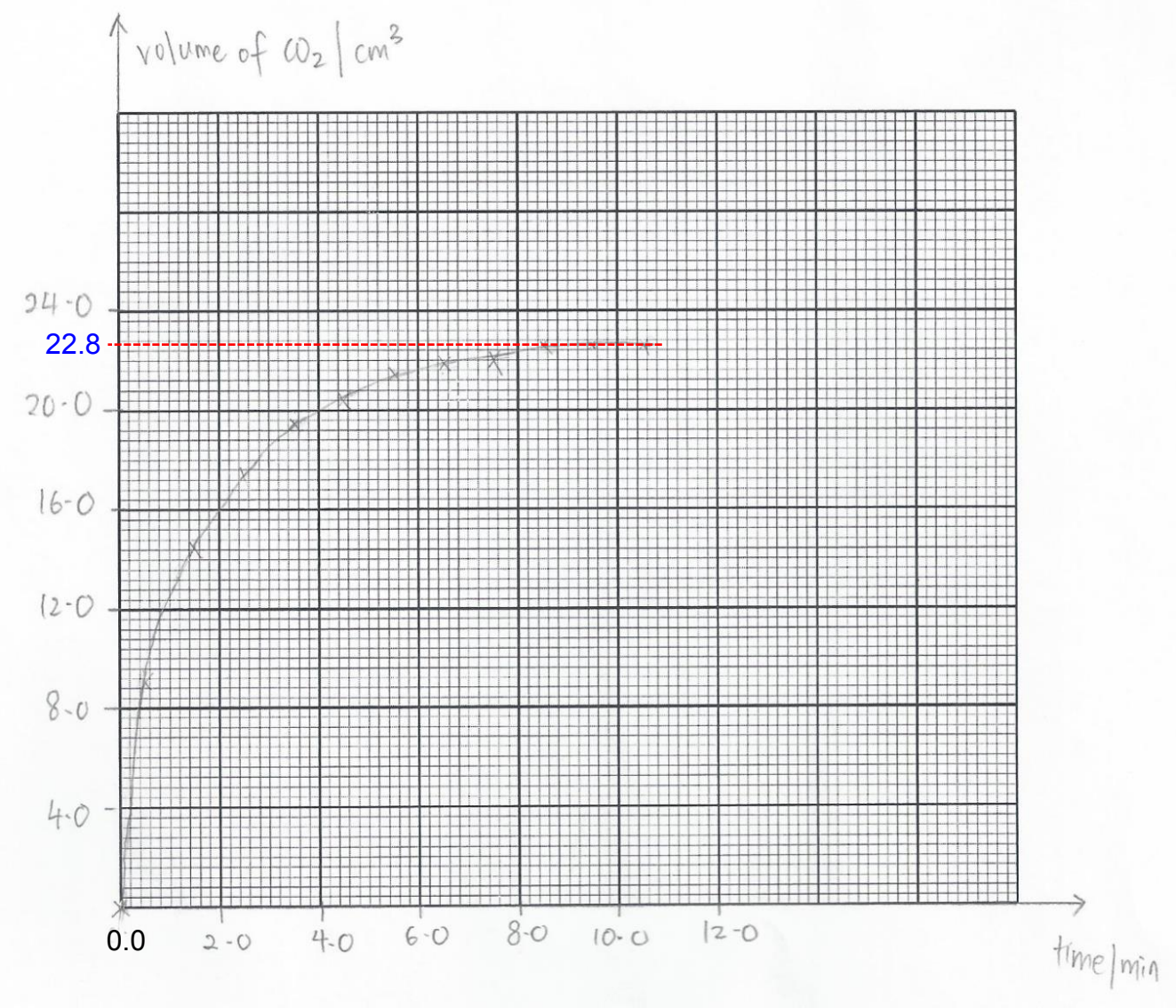
time / min	burette reading / cm^3	vol of CO_2 produced / cm^3
0.0	50.0	0.0
0.5	41.0	9.0
1.5	35.6	14.4
2.5	32.7	17.3
3.5	30.5	19.5
4.5	29.6	20.4
5.5	28.6	21.4
6.5	28.2	21.8
7.5	28.0	22.0
8.5	27.5	22.5
9.5	27.5	22.5
10.5	27.5	22.5

(ii) Plot on the grid below, a graph of volume of CO_2 on the y-axis, against time, *t*, on the x-axis.

Draw the most appropriate line, taking into account all your points.

Note: an appropriate line can be a curve since question did not specify it is a STRAIGHT line.

[4]



(iii) Write the balanced chemical reaction for the reaction between ethanedioate ions and manganate(VII) ions.

$5C_2O_4^{2-} + 2MnO_4^- + 16H^+ \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$
…….. [1]

(iv) Using your equation in **(iii)** and appropriate data from your graph, calculate a value for the amount of ethanedioate, $C_2O_4^{2-}$ ions, present in 20.0 cm^3 of **FA 1**.
[molar volume of gas = 24.0 dm^3 mol^{-1} at r.t.p.]

Amount of CO_2 = 22.8 / 24000 = 9.5×10^{-4} mol
Amount of $C_2O_4^{2-}$ = ½ x 9.5×10^{-4} = 4.75×10^{-4} mol

amount of ethanedioate, $C_2O_4^{2-}$ ions = 4.75×10^{-4} mol [1]

(v) Determine the concentration, mol dm^{-3}, of $C_2O_4^{2-}$ ions in **Q**.

In **FA 1**, $[C_2O_4^{2-}] = 4.75 \times 10^{-4} \div 20/1000 = 0.02375$ mol dm^{-3}
In **Q**, $[C_2O_4^{2-}] = 250/35.7 \times 0.02375 = 0.166$ mol dm^{-3}

concentration of $C_2O_4^{2-}$ ions in **Q** = 0.166 mol dm^{-3} [2]

(vi) Use your answer to **(v)** to calculate the M_r of the ethanedioate salt.

$[C_2O_4^{2-}] = [X_2C_2O_4] = 0.166$ mol dm^{-3}
$M_r = 43.0 \div 0.166 = 259.0$

M_r of the ethanedioate salt = 259.0 [1]

Hence deduce the identity of **X**.
Show your working.

[A_r: C, 12.0; O, 16.0; H, 1.0; Li, 6.9; Na, 23.0; K, 39.1; Rb, 85.5; Cs, 132.9; Fr, 223.0]

259.0 – (24.0+64.0) = 171.0
A_r of X = 171.0 /2 = 85.5
∴X is Rb.

X is Rb [2]

(b) **(i)** Another 25.0 cm^3 **FA 1** sample was analysed by titrating against acidified **FA 2** and the titre volume was found to be 28.50 cm^3.

Based on the results, calculate the concentration of $C_2O_4^{2-}$ in **FA 1**.

Amount of $C_2O_4^{2-} = \frac{5}{2}$ x Amount of MnO_4^-

$= \frac{5}{2} \times \frac{28.5}{1000} \times 0.02 = 1.425 \times 10^{-3}$ mol

$[C_2O_4^{2-}] = 1.425 \times 10^{-3} \div 0.025 = 0.0570$ mol dm^{-3}

concentration of $C_2O_4^{2-}$ in **FA 1** = 0.0570 mol dm^{-3} .....[1]

(ii) Using your answer to **(b)(i)**, determine the maximum volume of CO_2, at r.t.p., that could have been produced from 20.0 cm^3 of **FA 1**.

Amount of $CO_2 = 2 \times 0.020 \times 0.0570 = 2.28 \times 10^{-3}$ mol
Max volume of $CO_2 = 2.28 \times 10^{-3} \times 24000 = 54.7$ cm^3

maximum volume of CO_2 produced from 20.0 cm^3 of **FA 1** = 54.7 cm^3 .....[1]

(iii) Suggest a reason for the difference between the total volume of CO_2 you collected and the maximum volume of CO_2 calculated in **2(b)(ii)**.

The total volume of CO_2 collected is **less than** the calculated volume of CO_2. Some of the CO_2 gas has escaped to the atmosphere when **FA 2** is added to the conical flask.

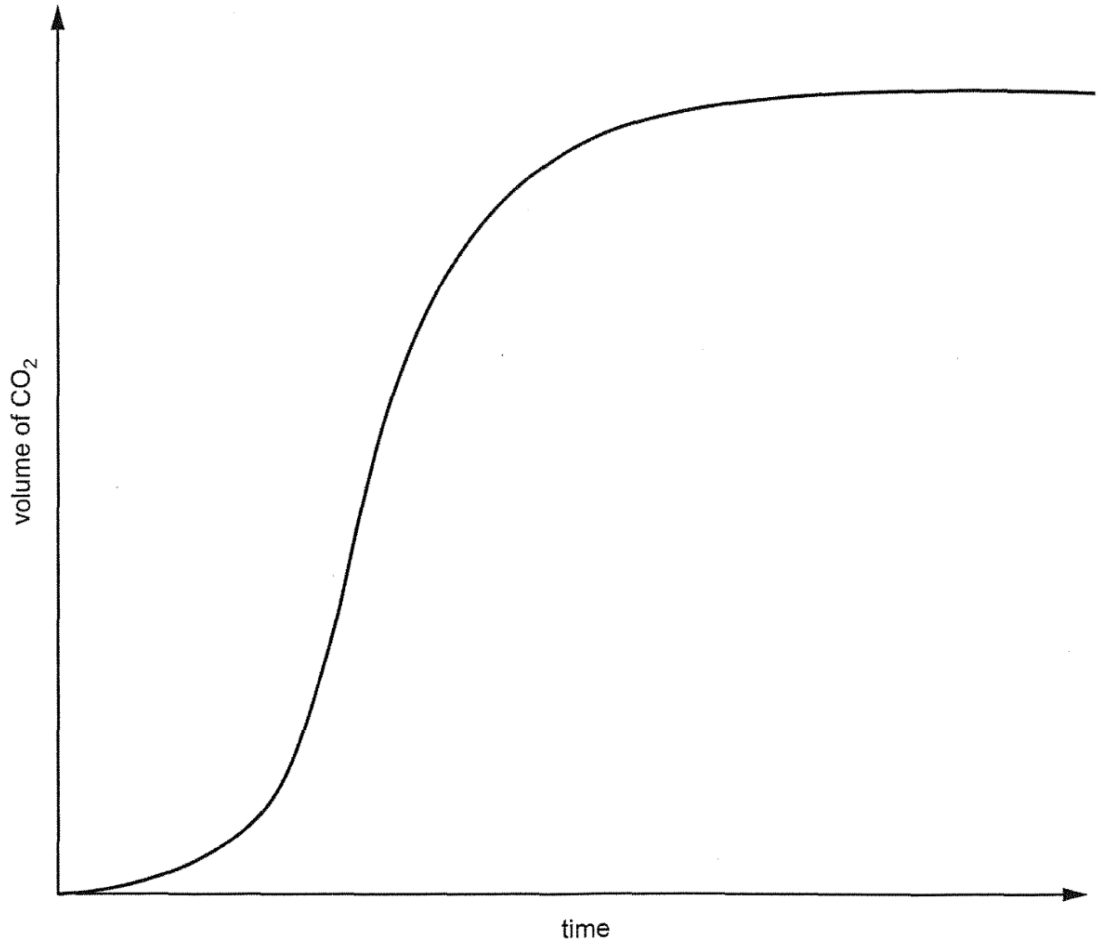
(iv) Suggest an improvement to this experiment that would overcome this problem.

Add **FA 2** through a dropping funnel and close the tap when 20.0 cm^3 of **FA 2** is added with some **FA 2** remaining in the funnel to create an air-tight environment.

(c) The presence of Mn^{2+} ions, which are produced in the reaction between MnO_4^- ions and $C_2O_4^{2-}$ ions, is thought to catalyse this reaction.

A student performed the experiment you performed in **2(a)(i)** but forgot to add **FA 4** to the mixture of **FA 1** and **FA 3** before adding the **FA 2**.

The student performed the experiment at the same temperature as your experiment and obtained the graph shown below.



Consider the **shape** of the graph above and your graph in **2(a)(ii)**.

Describe one major difference between their shapes. Suggest an explanation for your answer.

Difference:
The given graph shows a slow initial rate (less steep gradient) that increases after some time. For the plotted graph in **(a)(ii)**, the rate is fast from the start and gradually plateaus.

Explanation:
Without Mn^{2+} catalyst in the student's experiment, the reaction is slow at first. After some reaction has taken place, Mn^{2+} formed from the reaction catalyses the reaction and increases the rate so the gradient of the graph becomes steeper. For this experiment, Mn^{2+} catalyst is added at the beginning, hence rate is fast and plateaus off as the reactants are consumed.

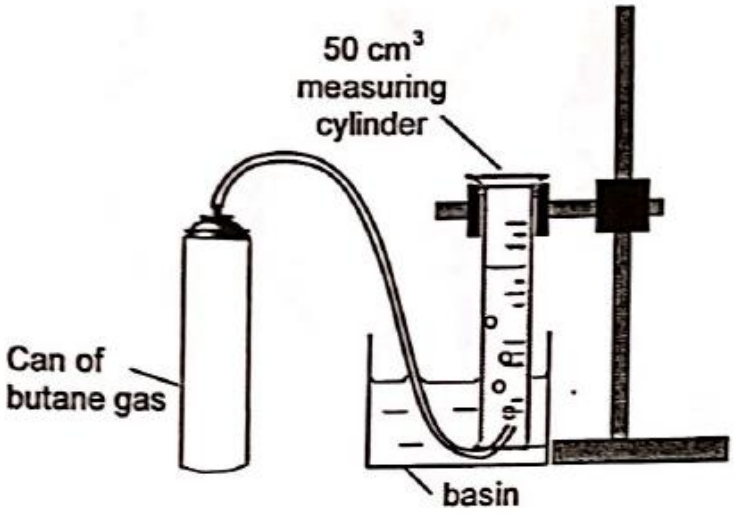
[Data Processing} : Gas Collection Practical 2

2. Determination of M_r of Butane

In this experiment, you are to use the ideal gas equation to find the relative molecular mass of a gas. Butane gas from a pressurised container is collected and the decrease in mass of the container is determined to allow relative molecular mass of butane to be calculated.

Procedure

1. Weigh a can of gas and record its mass in an appropriate manner.
2. Half fill the water container but fill the measuring cylinder with water fully.
3. Set up the retort stand next to the water container. Carefully invert the measuring cylinder full of water into the water container for gas collection. Clamp the measuring cylinder in position.
4. Insert the open end of the rubber tubing into the measuring cylinder. Attach the other end of the tubing with an adaptor to the gas can nozzle.
5. The liquid level inside the measuring cylinder should be on the graduated scale. If it is not, introduce some air bubbles into the cylinder using a dropper. Remember that the scale is now upside down. Record the initial measuring cylinder reading.



6. Lightly depress the gas nozzle to release some gas into the measuring cylinder and collect about 40 cm^3 of gas. Record the final measuring cylinder reading.
7. Reweigh the can of gas, making sure that it is dry.
8. Record the temperature of the laboratory.

Results

Table 1: Mass readings

mass of can of gas before experiment /g	239.409
mass of can of gas after experiment /g	239.304
Mass of butane gas used /g	0.105

Table 2: Volume readings

Initial measuring cylinder reading/ cm^3	8.0
Final measuring cylinder reading /cm^3	50.0
Volume of butane gas / cm^3	42.0

Temperature of laboratory = 30.5 ºC

(a) By using the readings that you have obtained in this experiment and the following equation, determine the relative molecular mass of butane.

$$pV = nRT$$

$M = \frac{(0.105)(8.31)(30.5+273)}{(101000)(42\ x\ 10^{-6})} = 62.4\ gmol^{-1}$

$M_r = 62.4$

62.4

M_r of butane = …….. [2]

(b) **(i)** Compare your calculated M_r of butane in **(a)** with the theoretical value. Give **one** reason that could account for the difference between your calculated M_r of butane and the theoretical value.

Theoretical M_r of butane is 58.0 which is smaller than the calculated 62.4.

Reason that resulted in a larger M_r:

- Due to intermolecular forces of attraction between butane, the volume measured is less than expected.
- butane gas trapped in the water column are not collected and measured, resulting the V_{butane} measured to be less than expected.
- Volume of butane that is used to displace the water column in the delivery tube cannot be measured, resulting the V_{butane} measured to be lesser than expected.

A smaller measured volume resulted in a larger calculated M_r.

(iii) Identify **one** error in the procedure that affects the reliability of the results. Suggest an improvement that could be made to reduce the error you have identified.

1. Use of a less precise instrument, i.e. measuring cylinder to collect butane, which results in higher percentage error due to measurement.

$$\% \text{ error} = \frac{\pm 0.5}{42.0} \times 2 \times 100\% = \pm 2.38\%$$

2. Experiment is not repeated to ensure consistency.

Improvement for Error (1):

A more precise instrument such as a burette should be used.
% error using burette = $\frac{\pm 0.05}{42.00}$ x 2 x 100% = ±0.24%

Improvement for Error (2):
Repeat the experiment until values of $\frac{V}{m}$ are within 5 % and use an average of the consistent gas volumes for subsequent calculations.

Data Processing

[Data Processing] ; Gravimetric Analysis Practical 1

1. Determination of the formula of magnesium oxide

In this experiment, the formula of magnesium oxide will be determined by heating magnesium wire in air to form magnesium oxide. The increase in mass at the end of the heating process is due to the oxygen that has combined with the magnesium to form magnesium oxide.

(a) You are to follow the practical procedure exactly and record your measurements in a suitable format. You should carefully consider the most effective way of handling the materials and apparatus, so as to obtain the best result.

- Weigh a crucible and lid.
- Clean about 2.5 cm of magnesium ribbon using a sandpaper.
- Place the magnesium ribbon in the crucible and weigh this together with the lid.
- Place the crucible on a wire gauze on a tripod and heat strongly for 2 minutes. Lift the lid very slightly to let more oxygen in but immediately replace it to prevent magnesium oxide from escaping.
- Do this until the magnesium no longer flares up. Remove the lid and heat strongly for five minutes. Allow the crucible to cool on a heat-proof mat and then re-weigh with lid.
- Repeat the heating, cooling and weighing cycle until constant mass of ±0.010 g is achieved.

Results

Table 1: Mass of Magnesium ribbon

Mass of crucible, lid and magnesium ribbon /g	5.225
Mass of crucible and lid /g	5.025
Mass of magnesium ribbon /g	0.200

Table 2: Mass of Magnesium oxide

Mass of crucible, lid and content after 1st heating /g	5.230
Mass of crucible, lid and content after 2nd heating /g	5.335
Mass of crucible, lid and content after 3rd heating /g	5.359
Mass of crucible, lid and content after 4th heating /g	5.367
Mass of crucible and lid /g	5.025
Mass of magnesium oxide /g	0.342

(b) **(i)** Determine the mass of oxygen that has combined with magnesium.

Mass of oxygen = 0.342 – 0.200 = 0.142 g

0.142 g

mass of O_2 = ……..…… [1]

(ii) Hence, determine the formula of magnesium oxide.

No. of moles of magnesium = $\frac{0.200}{24.3}$ = 8.23 x 10^{-3} mol

No. of moles of oxygen = = $\frac{0.142}{16.0}$ = 8.875 x 10^{-3} mol

simplest ratio of Mg : O is 1:1

Therefore, formula of magnesium oxide is MgO.

formula = MgO [2]

(c) **(i)** Explain the purpose of **step 2** in the procedure in **(a)**.

This is to **remove the layer of MgO**, thus the mass of the magnesium ribbon used is the mass of pure Mg. Calculated amount of Mg, hence calculated Mg : O mole ratio is thus more accurate.

(ii) A student repeated the experiment using the procedure in **(a)** but did not carry out **step 6**. Explain the effect on his determination of the formula of magnesium oxide.

Repeating the heat-cool-weigh process until constant mass ensures that the **oxidation of magnesium is complete**. If this is not done, the oxidation of the magnesium may not go to completion. The **mass of magnesium oxide would be less than expected**, mass of O calculated be less than expected and **the ratio of Mg : O would be greater than 1:1.**

[Data Processing] ; Gravimetric Analysis Practical 2

2. Determination of the number of molecules of H_2O in hydrated barium bromide

The water of crystallisation in hydrated barium bromide is lost when heated.

$$BaBr_2.xH_2O\ (s) \rightarrow BaBr_2\ (s) + x\ H_2O\ (g)$$

In this experiment, you are to heat samples of varying mass of barium bromide and to determine the number of molecules of H_2O in hydrated barium bromide by plotting a graph of the mass of barium bromide against the mass of the anhydrous barium bromide left after heating.

(a) Procedure

A student weighed a clean, dry crucible and transferred 0.40 g of hydrated barium bromide to it. The crucible and its contents were heated until a constant mass has been reached (heating-cooling-weighing method). This mass was then recorded.

The student then repeated the experiment using different masses of hydrated barium bromide.

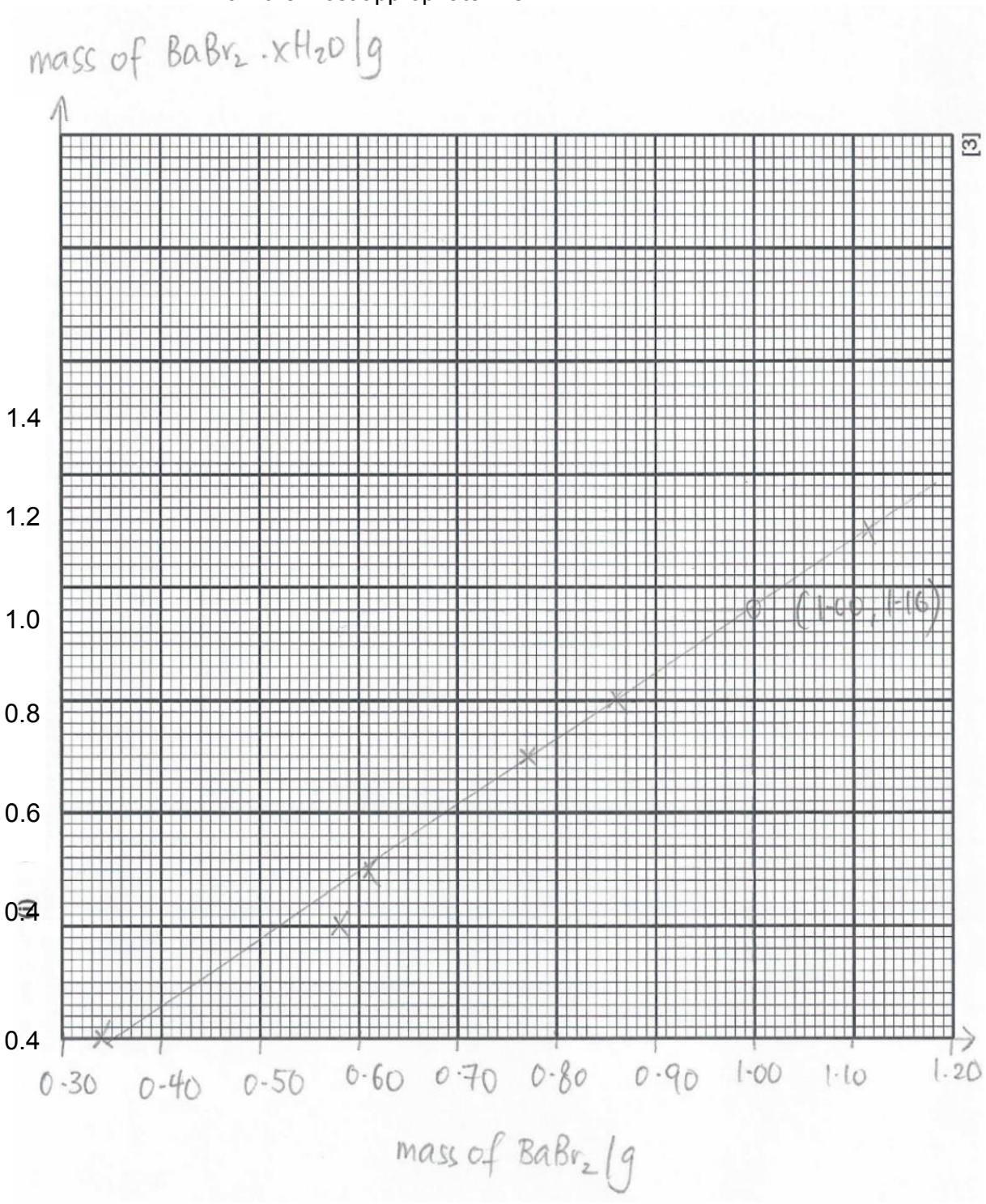
For each experiment the student recorded the original mass of the hydrated barium bromide and the mass of anhydrous barium bromide left after heating. The results are as shown below.

Results

Experiment	mass of $BaBr_2.xH_2O$ / g	mass of $BaBr_2$ / g
1	0.40	0.34
2	0.60	0.58
3	0.70	0.61
4	0.90	0.77
5	1.00	0.86
6	1.30	1.13

(b) **(i)** Plot on the grid below, a graph of the mass of barium bromide on the y-axis, against the mass of the anhydrous barium bromide on the x-axis.

Draw the most appropriate line.



[4]

(ii) Use this graph to determine the mass of hydrated barium bromide which forms 1.00 g of anhydrous barium bromide.

1.16 g

mass = …… [1]

(iii) Calculate the number of moles of $BaBr_2$ present in 1.00 g of anhydrous barium bromide.
[*Ar*: Ba, 137.3; Br, 79.9]

No. of moles of $BaBr_2 = \frac{1.00}{297.1} = 3.37 \times 10^{-3}$ mol

No. of moles of $BaBr_2$ = 3.37 x 10^{-3} mol ..... [1]

(iv) Using your answer to **(ii)** and **(iii)**, calculate the M_r of hydrated barium bromide.

$n(BaBr_2.xH_2O) = n(BaBr_2) = 3.37 \times 10^{-3}$ mol

Molar mass of $BaBr_2.xH_2O = \frac{1.16}{3.37 \times 10^{-3}} = 344.6$ $gmol^{-3}$

M_r of hydrated barium bromide = 344.6

M_r of $BaBr_2.\mathbf{x}H_2O$ = 344.6 ..... [1]

Hence calculate the value of **x** in $BaBr_2.\mathbf{x}H_2O$. Give your answer to the nearest whole number.
[*Ar*: H, 1.0; O, 16.0; Ba, 137.3; Br, 79.9]

137.3 + 79.9 x 2 + 18x = 344.6
x = 2.64
x = 3 (whole number)

x is 3 .....[2]

(c) Considering your answer obtained in **(b)(iv)** above, comment on the results obtained by the student. Is your line of best fit good enough to be used with confidence?

The value of x deviates greatly from a whole number. This shows that the line of best fit cannot be used with confidence.

(d) A databook value for the M_r of hydrated barium bromide is 333.1. Calculate the difference between the M_r value obtained from the student's data and the databook value. Express this difference as a percentage of the databook value.

Difference in M_r = 344.6 – 333.1 = 11.5
Percentage difference = $\frac{11.5}{333.1}$ x 100 % = 3.45 %

percentage difference = 3.45 % ..... [1]

(e) **(i)** Explain why there is a need to heat the crucible and its content to constant mass.

The constant mass indicates that **all of the water of crystallisation has been removed from the hydrated barium bromide**.

(ii) Suggest an explanation why the hydrated barium bromide should not be stirred during heating.

This will result in a loss of mass as some solid could spill out and some will stick to the stirrer.

[Data Processing] ; Gravimetric Analysis Practical 3

3 Determination of the the M_r of an unknown metal carbonate.

.

The amount of carbon dioxide evolved when a metal carbonate, X2CO3, reacts with an excess of an acid is directly proportional to the mass of the carbonate used. The relative molecular mass of X2CO3 can hence be determined by conducting a simple experiment.

Procedure

1. The mass of a weighing bottle containing X_2CO_3 is measured.
2. Using a suitable measuring cylinder, 50.0 cm^3 of 2.0 mol dm^{-3} hydrochloric acid (in excess) is transferred to a 250 cm^3 conical flask.
3. A cotton wool plug is placed in the neck of the flask and the flask + acid is weighed.
4. The weighed X_2CO_3 is added to the acid in the flask and the cotton wool plug is quickly replaced in the neck of the flask to prevent any loss of acid spray.
5. The mass of the empty weighing bottle is measured.
6. When the reaction in the flask has stopped, the flask is left for 10 minutes to allow carbon dioxide to diffuse from the flask.
7. The mass of the flask and its contents after the reaction are measured.

Results

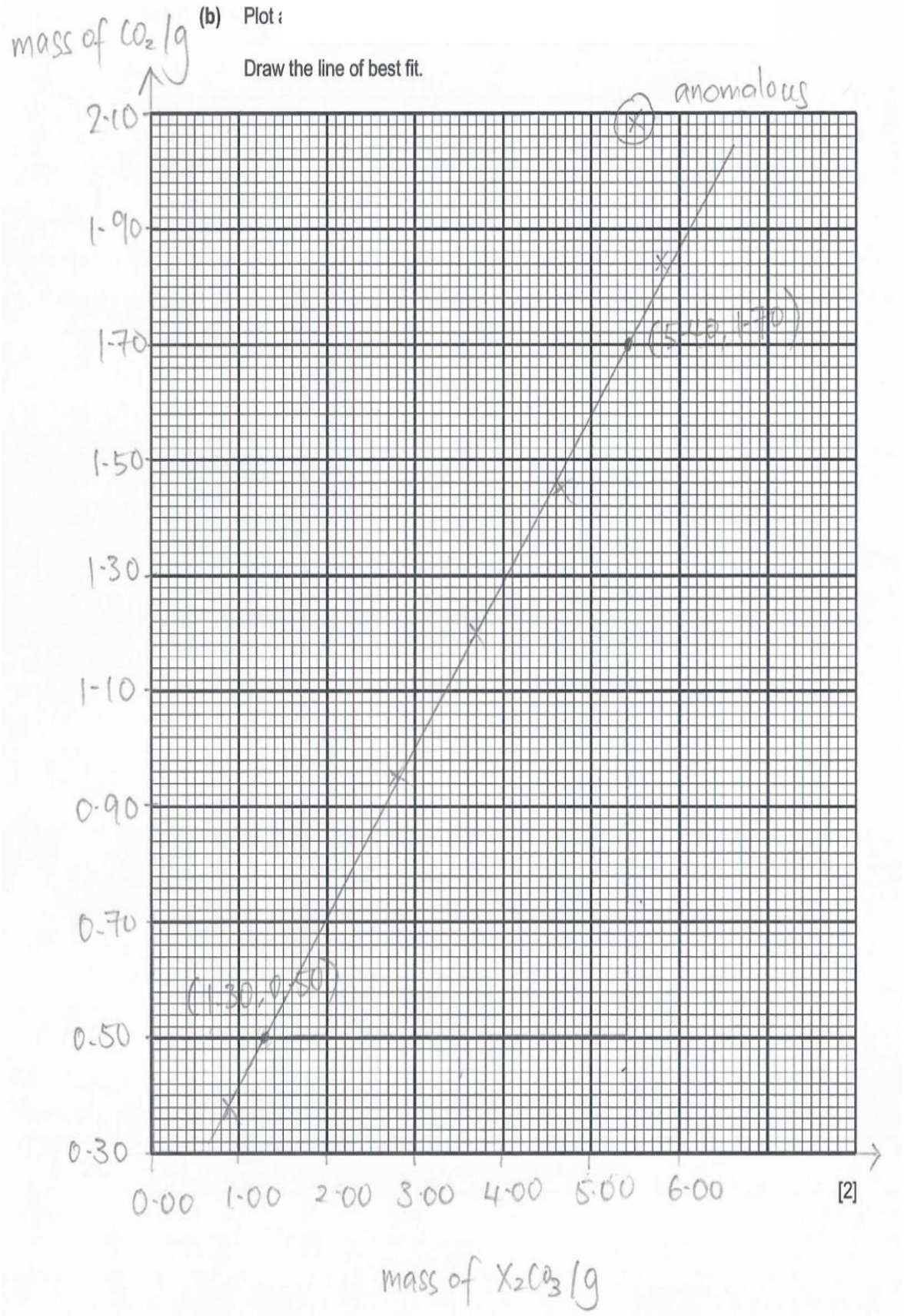
A	**B**	**C**	**D**	**E (A – B)**	**F (C + E – D)**		
mass of weighing bottle + X2CO3 / g	mass of empty weighing bottle / g	mass of flask + acid before the reaction / g	mass of flask + solution after the reaction / g	mass of X_2CO_3 used / g	mass of CO_2 given off / g		
14.29	11.48	221.35	223.21	2.81	0.95		
16.41	11.76	209.71	212.91	4.65	1.45		
12.24	11.34	210.45	210.97	0.90	0.38		
16.77	11.27	214.38	217.80	5.50	2.08		
16.48	10.68	211.63	215.59	5.80	1.84		
14.85	11.15	217.18	219.68	3.70	1.20		

(a) Process the results in the table to calculate both the mass of X_2CO_3 used and the mass of CO_2 given off. Record these values in the additional columns of the table. You may use some or all of these columns.

Label the columns you use, including the units and an equation to show how the value is calculated. You may use the column headings **A** to **H** in these equations, e.g. = **C** – **B**.(Remember that the gas is lost from the flask after the weighed carbonate is added to the weighed acid.) [3]

(b) Plot a graph of mass of CO_2 / g against mass of X_2CO_3 / g.

Draw the line of best fit.



(c) Circle **one** clearly anomalous point on your graph. By reference to the description of the experiment, suggest an explanation for this anomaly.

The data for 5.50 g of X_2CO_3 is anomalous as the mass loss of CO_2 is significantly higher than expected (far from the best fit line).

Some acid may have escaped from the flask as acid spray when the cotton wool was removed to add the solid.

(d) **(i)** Clearly mark two points on your line of best fit suitable to be used to determine the gradient of the line. Write the coordinates of the points you have selected below.
Use these points to determine the gradient of the line.

(5.40, 1.70) & (1.30, 0.50)

Gradient = (1.70 – 0.50) / (5.40 – 1.30)
= 0.293

gradient = 0.293 [1]

(ii) Use the gradient you have calculated and the information below to calculate the M_r for X_2CO_3.

$X_2CO_3(s) + 2H^+(aq) \rightarrow 2X^+(aq) + CO_2(g) + H_2O(l)$

From chemical equation,

$n(CO_2) = n(X_2CO_3)$

mass of CO_2 (y-axis) / M_r of CO_2 = mass of X_2CO_3 (x-axis) / M_r of X_2CO_3

$y = mx$

$\frac{\text{Mr of } CO_2}{\text{Mr of } X_2CO_3} = m = 0.293$

$Mr(CO_2) = 44.0$

$\therefore Mr \text{ of } X_2CO_3 = 150.2$

M_r for X_2CO_3 = 150.2 [1]

(f) Explain how each of the following changes to the experiment would affect the gradient of the graph obtained from the experiment.

(i) The experiment is carried out at a higher temperature.

No change. The mass of CO_2 lost does not depend on temperature.

(ii) The experiment is carried out with a different carbonate, $\mathbf{Y}_2CO_3$, which has a lower M_r than $\mathbf{X}_2CO_3$.

A smaller M_r of $\mathbf{Y}_2CO_3$ would mean a **larger gradient** since gradient of the graph plotted = M_r of CO_2 / M_r of $\mathbf{Y}_2CO_3$.

(g) Use your knowledge of acid-base chemistry to suggest a more appropriate way by which the M_r of a soluble metal carbonate might be determined.

A standard solution of the metal carbonate can be prepared by dissolving it in deionised water using a graduated flask.

Aliquots of this solution can then be titrated against acid of known concentration to determine the concentration of carbonate ions.

[Data Processing] ; Gravimetric Analysis Practical 4

4. Determination of formula of a hydrated basic carbonate

The approximate formula for a hydrated basic carbonate is $CuCO_3.Cu(OH)_2.H_2O$. When heated, the basic copper(II) carbonate decomposes.

$$CuCO_3.Cu(OH)_2.H_2O(s) \rightarrow 2CuO(s) + CO_2(g) + 2H_2O(g)$$

FA 1 is powdered basic copper(II) carbonate, a hydrated mixture of copper(II) carbonate and copper(II) hydroxide.

Procedure

Record all weighings in an appropriate format in the space below.

1. Weigh and record the mass of an empty boiling-tube.
2. Tip the contents of the tube labelled **FA 1** into the weighed boiling-tube. Reweigh and record the total mass of the boiling-tube and **FA 1**.
3. Heat **FA 1** in the boiling-tube **very gently** until the vigorous decomposition of the copper carbonate has stopped; then heat more strongly for 1 to 2 minutes. **Take care not to lose any solid from the tube during the initial heating.**
4. Warm the upper parts of the boiling-tube to remove any water that may have condensed while heating the carbonate.
5. Place the hot tube on a heat-proof mat and leave to cool.
6. Reweigh the boiling-tube and the residual copper(II) oxide.
7. Repeat the heating, cooling and weighing process until a constant mass of ±0.010 g is achieved.

Results

Mass of boiling tube and content after 1st heating /g Mass of boiling tube and content after 2nd heating /g Mass of boiling tube and content after 3rd heating /g Mass of boiling tube and content after 4th heating /g	6.951 6.912 6.869 6.860
Mass of empty boiling tube /g	6.631
Mass of boiling tube + **FA 1** / g	6.984
Mass of **FA 1** / g	0.353
Mass of residual copper oxide /g	0.229

(a) Calculate the loss in mass during the experiment as a percentage of the mass of solid heated.

mass loss = 0.353 – 0.229 = 0.124 g

% mass loss = 0.124 / 0.353 x 100 = 35.1 %

35.1 %

percentage loss in mass = …… [1]

(b) The theoretical loss in mass is 33.5 %.

The proportions of $CuCO_3$ and $Cu(OH)_2$ in the basic carbonate can vary from the 1:1 ratio given in the formula.

Make use of the following information to account for the difference between the value you have calculated in **(a)** and the theoretical percentage loss in mass.

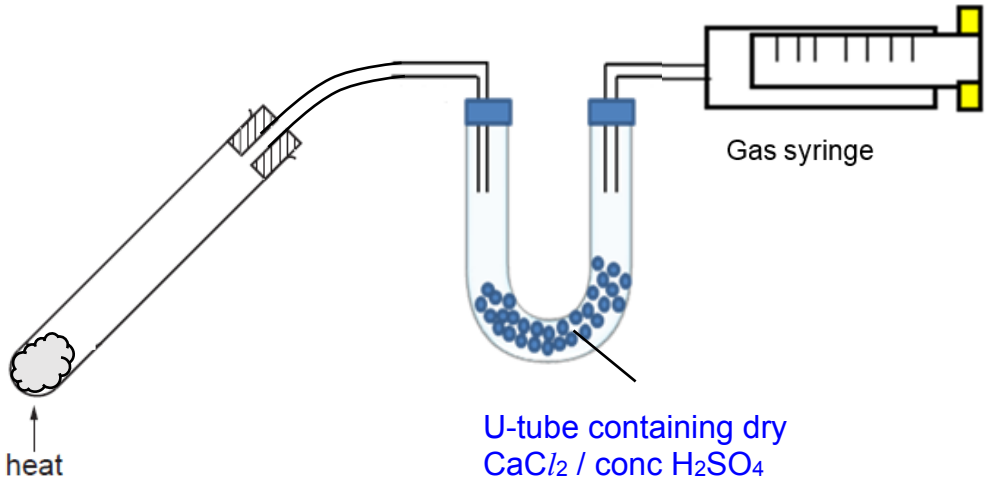
1 mol $CuCO_3(s) \rightarrow$ 1 mol $CO_2(g)$
1 mol $Cu(OH)_2(s) \rightarrow$ 1 mol $H_2O(g)$
Assume that 1 mol of any sample of the solid basic carbonate contains 1 mol H_2O.
[*Mr*: CO_2, 44.0; H_2O, 18.0]

Since the experimental mass loss is more than theoretical value, the mass loss is more than expected. This suggests that the loss of CO_2 is more than expected, hence **the proportion of $CuCO_3$: $Cu(OH)_2$ is more than 1:1**.

Note:
If mixture contains a larger proportion by mass of $CuCO_3$, the mass loss will be greater than expected.

(c) Add to the diagram below additional standard laboratory apparatus that would enable you to collect and measure the volume of carbon dioxide evolved in the experiment.

Ensure that your apparatus does not also collect and measure any of the water vapour evolved.



$CaCl_2$ / conc H_2SO_4 is a drying agent, which removes water vapour from the gas mixture.

[2]

Data Processing

[Data Processing] : Thermochemistry Practical 1

1. Determination of the enthalpy change of combustion, $\Delta H_{combustion}$, of a fuel.

In this experiment, you are required to determine the enthalpy change of combustion of a fuel **A**, by first calibrating the calorimeter using a tea-light candle.

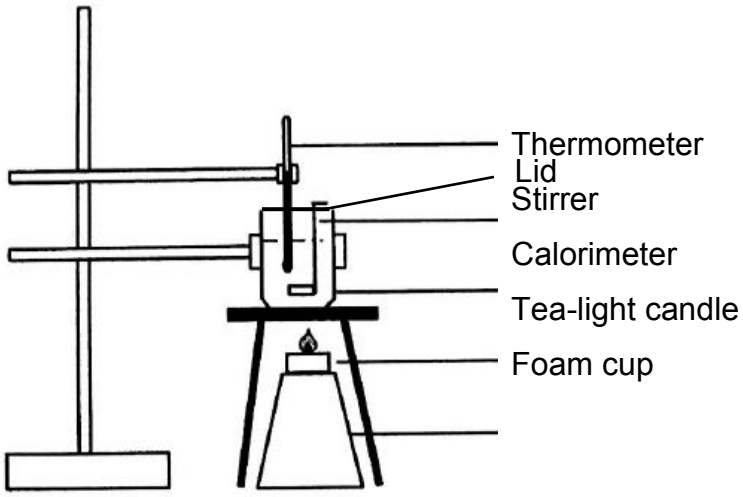
The main compound that is burnt during combustion of tea-light candles is paraffin wax. The paraffin wax or paraffinic hydrocarbons used usually consists of straight chained or branched alkanes with formula C_nH_{2n+2} where n ranges from 18 to 30.

In the first set up, the heat evolved from the combustion of paraffin wax is used to heat up a fixed volume of water.

In the second set-up, an unknown fuel **A** undergoes combustion in a fuel lamp. This process is exothermic and heats up the water in the calorimeter. By measuring the temperature rise and using the heat capacity of the calorimeter, the enthalpy change of combustion of fuel **A** can be calculated.

(a) Calibration of calorimeter using tea-light candles

1. Set up the apparatus as shown in the diagram below.



2. Measure the mass of the candle and record its mass.
3. Fill the calorimeter with 100 cm^3 of water using a 100 cm^3 measuring cylinder. Record the initial temperature of the water using a 1 ^oC division thermometer.
4. Light the wick of the candle and place it under the calorimeter. Stir the contents gently.
   *Note: The candle should be placed under the calorimeter such that the tip of the wick is approximately 2 cm from the base of the calorimeter.
5. Extinguish the flame when a rise of about 5 ^oC is reached and record the maximum temperature reached after the flame has been extinguished.
6. Allow the candle to cool sufficiently and reweigh the burnt candle, recording the new mass of the candle. Record your results in the table below.

Table 1: Calibration of calorimeter using tea-light candle

Mass of tea-light candle before combustion /g	12.950
Mass of tea-light candle after combustion /g	12.602
Mass of wax burnt /g	0.348
Initial temperature of water /ºC	28.0
Maximum temperature reached by water / ºC	33.0
ΔT / ºC	+5.0

(b) Finding the enthalpy change of combustion of fuel A

7. For the same setup shown above, use the fuel lamp containing fuel **A** in place of tea-light candle.
8. Repeat steps **2** to **6** using the lamp.
9. Record your results in the table below.

Table 2: Combustion of fuel A

Mass of fuel lamp before combustion /g	171.387
Mass of fuel lamp after combustion /g	170.527
Mass of fuel burnt /g	0.860
Initial temperature of water /ºC	28.0
Maximum temperature reached by water / ºC	33.5
ΔT / ºC	+5.5

(c) (i) Given that heat energy, q = CΔT, calculate C, the heat capacity of the calorimeter.

Relevant data:

- $\Delta H_{combustion}$ of paraffin wax (n = 22) = −11.0 kJ mol^{-1}
- M_r of fuel **A** = 46.0

No. of moles of $C_{22}H_{46}$ = $\frac{0.348}{310.0}$ = 0.001123 mol

Heat, q = 11000 x 0.001123 = 12.35 J
heat capacity of calorimeter, C = q/ ΔT = $\frac{12.35}{5.0}$ = 2.47 J K^{-1}

heat capacity of calorimeter = 2.47 J K^{-1} ..... [1]

(ii) Calculate the enthalpy change of combustion of fuel **A**

heat, q = C x ΔT = 2.47 x 5.5 = 13.59 J
No of moles of fuel **A** = $\frac{0.860}{46.0}$ = 0.01870 mol

$\Delta H_{combustion} = -\frac{q}{n_{limiting\ reagent}} = -\frac{13.59}{0.01870}$ = −727 J mol^{-1}

enthalpy change of combustion of fuel **A** = −727 J mol^{-1} ..... [1]

(d) Identify **two** sources of error in the procedure and measurement that affect the reliability of the results. Explain how each error affects the results obtained and suggest a way to reduce each error.

Error 1:

During cooling of fuel lamp, fuel **A** continues to evaporate due to high temperature. Hence the mass of fuel measured is higher than actual, which results in enthalpy change of combustion fuel **A** being less exothermic / having a smaller magnitude. To overcome this, extinguish the fuel lamp with a lid or cap. Measure the mass of the fuel lamp with the lid / cap when measuring its initial and final mass.

Error 2:

The use of less precise 1°C division thermometer to measure the temperature of water in the calorimeter. This leads to a large percentage error due to measurement which results in enthalpy change of combustion fuel **A** being greater or smaller. To overcome this, use a 0.2 °C division thermometer.

(e) Assuming the same mass of fuel **A** is used, explain the effect on ΔT measured if the following change is made to the procedure.

(i) The calorimeter is filled with 50 cm^3 of water instead of 100 cm^3.

The same amount of heat is evolved from combustion of fuel **A** is absorbed by a smaller mass of water. Since $q = mc\Delta T$, ΔT is greater in magnitude.

(ii) The calorimeter is changed from a copper container to a glass beaker.

Glass has a greater specific heat capacity compared to copper. The heat capacity of the calorimeter will be greater, hence the same amount of heat evolved from combustion of fuel **A** results in ΔT that is smaller in magnitude.

[Data Processing] ; Thermochemistry Practical 2

2. Determination of ΔH_{rxn} using Hess's Law

Copper is an unreactive metal that does not react directly with dilute acids. It is therefore necessary to find the enthalpy change of reaction for two reactions that do occur so that Hess' Law can be used to determine the enthalpy of reaction.

Reaction 1: $Mg(s) + H_2SO_4(aq) \rightarrow MgSO_4(aq) + H_2(g)$

Reaction 2: $Mg(s) + CuSO_4(aq) \rightarrow MgSO_4(aq) + Cu(s)$

In this experiment, you are required to determine the enthalpy change of reaction, ΔH, for the reaction shown below.

$$Cu(s) + H_2SO_4(aq) \rightarrow CuSO_4(aq) + H_2(g)$$

You are given the following:

FA 1 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4
FA 2 is magnesium powder, Mg in a stoppered tube
FA 3 is 1.00 mol dm^{-3} copper(II) sulfate, $CuSO_4$.
FA 4 and **FA 5** are magnesium powder, Mg in stoppered tubes

(a) Determining the enthalpy change for Reaction 1

Read through the method before you start any practical work and prepare suitable tables for your results.

1. Weigh the stoppered tube containing **FA 2**. Record the mass in an appropriate manner.
2. Support the plastic cup in the 250 cm^3 beaker.
3. Use a 50 cm^3 measuring cylinder to transfer 50.0 cm^3 of **FA 1** into the plastic cup.
4. Measure the temperature of **FA 1** in the plastic cup, using a 0.2 °C division thermometer and start the stop clock. Record this temperature as being the temperature at time = 0.
5. Measure, and record, the temperature of this **FA 1** every half minute for 2 minutes.
6. At time = 2½ minutes, add the **FA 2** to the acid and stir carefully to reduce acid spray.
7. Measure the temperature of the mixture in the cup at time = 3 minutes and then every half a minute up to time = 7 minutes. Continue stirring occasionally throughout this time.
8. Weigh the stoppered tube that had contained **FA 2**. Record the mass.
9. Calculate and record the mass of **FA 2** added to the sulfuric acid.
10. Rinse the plastic cup with water and shake to dry.

Results

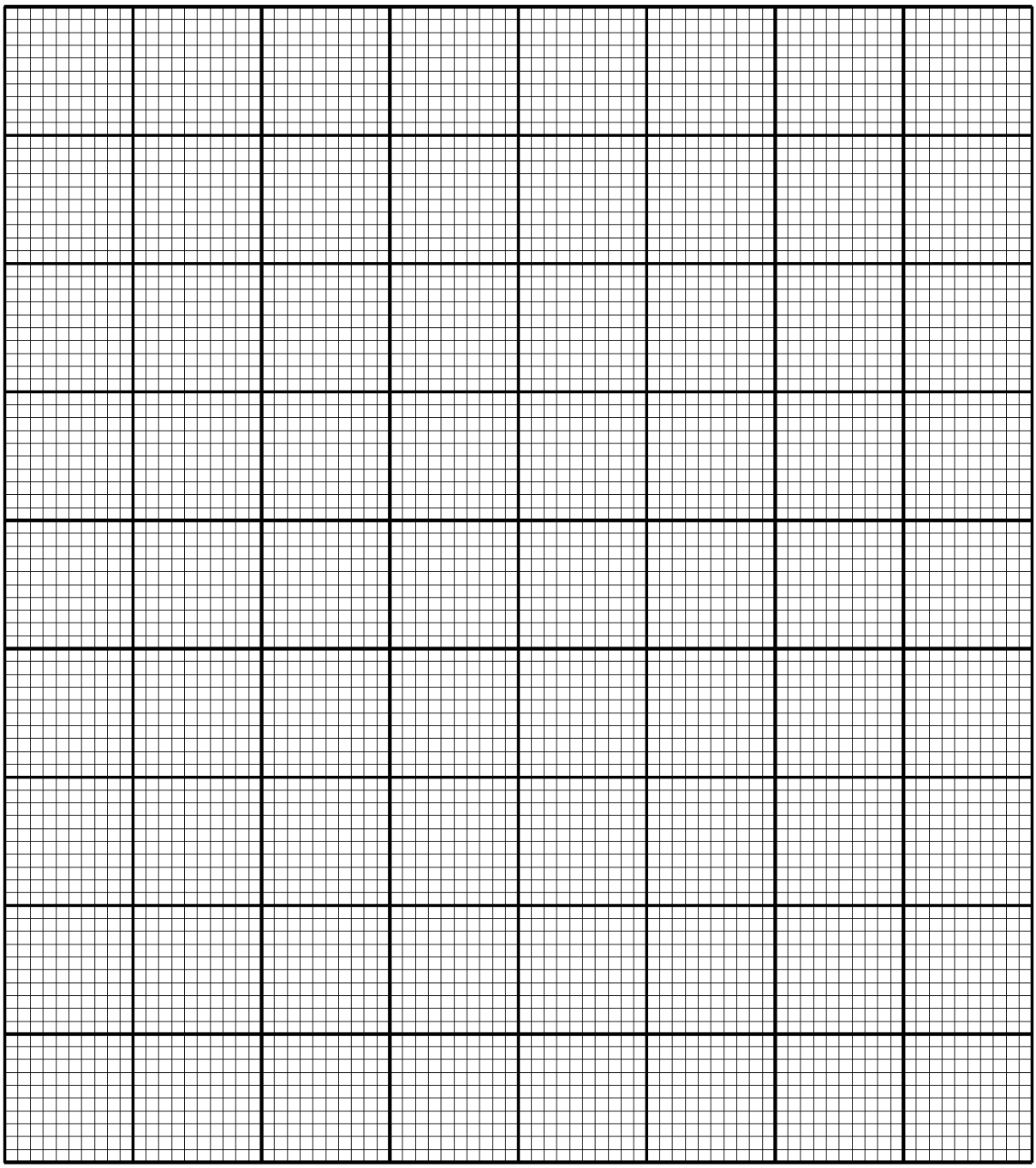
Table 1: Mass of FA2

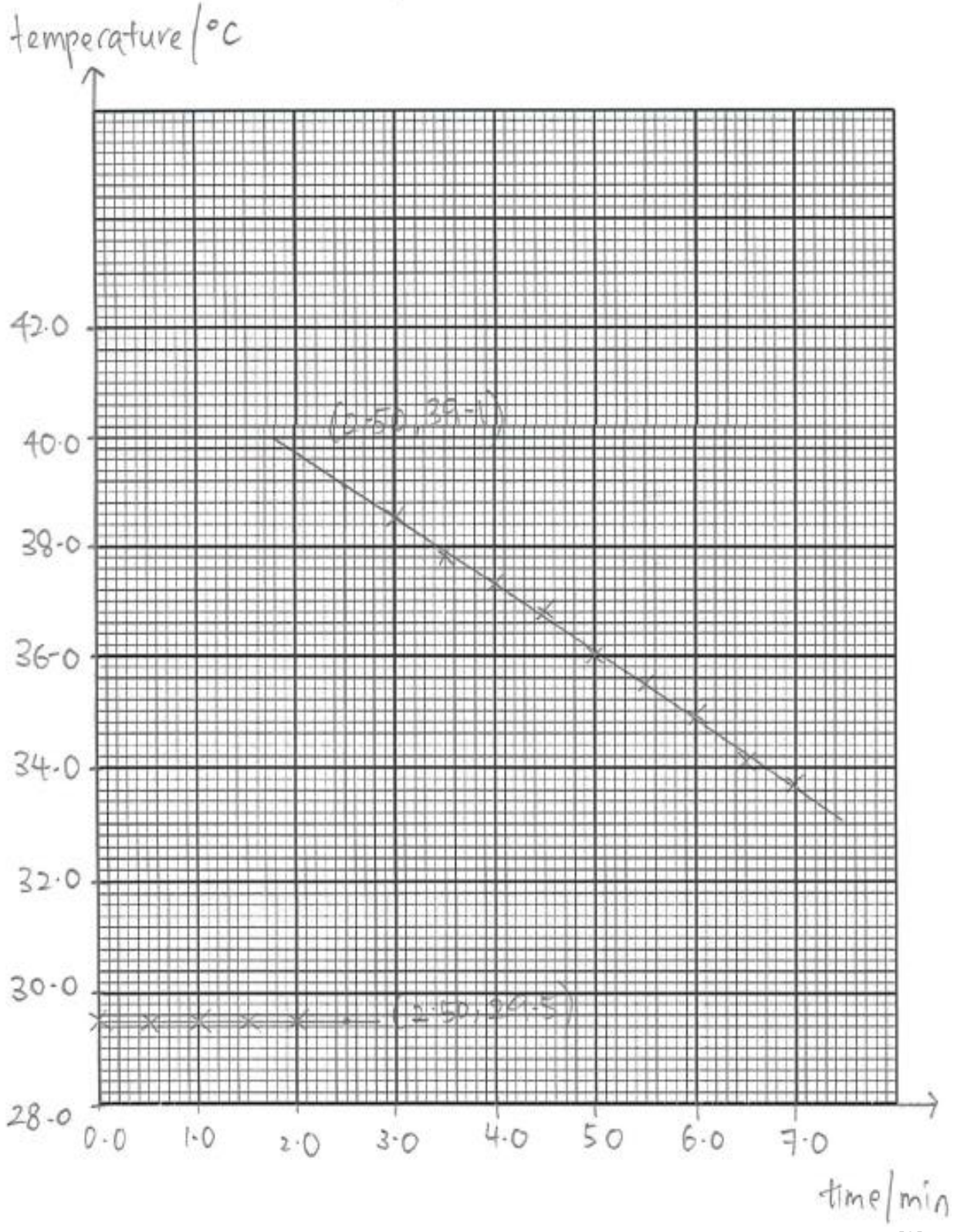
Mass of stoppered tube and **FA 2** /g	10.655
Mass of stoppered tube and residual **FA 2** /g	10.548
Mass of **FA 2** used /g	0.107

Table 2: Temperature recordings

Time/ min	Temperature/ ºC	Time/ min	Temperature/ ºC
0.0	29.5	**4.0**	37.3
0.5	29.5	**4.5**	36.8
1.0	29.5	**5.0**	36.0
1.5	29.5	**5.5**	35.5
2.0	29.5	**6.0**	34.9
2.5	-	**6.5**	34.1
3.0	38.5	**7.0**	33.7
3.5	37.8		

(i) On the grid below plot a graph of temperature (*y*-axis) against time (*x*-axis).





[3]

(ii) Complete the graph by inserting **two**, **straight** lines of best fit:
- one to show the temperature up to time = 2½ minutes,
- one to show the temperature after time = 2½ minutes

(iii) From your graph, use the two straight lines of best fit to calculate the change in temperature at time = 2½ minutes.

max T at 2.5 min can be 39.1 °C (follow your plotted graph)

min T at 2.5 min can be 29.5 °C (follow your plotted graph)

+9.6 °C

temperature change = ... [1]

(b) **(i)** In the reaction in **(a)**, sulfuric acid was in excess. Without carrying out any additional tests, what observation could you have made during your experiment to confirm this?

All the magnesium / solid dissolved / disappeared or all solid / Mg has gone / been used up or no solid / Mg left.

(ii) Calculate the heat change that occurred during the reaction in **(a)**.
[Assume that 4.2 J is needed to raise the temperature of 1.0 cm^3 of solution by 1.0 °C.]

q = mcΔT = 50 x 4.2 x 9.6 = 2016 = 2.02 kJ

2.02 kJ

heat change = ... [1]

(iii) Use your answer to **(ii)** to calculate the enthalpy change, in kJ mol^{-1}, for the reaction between sulfuric acid and magnesium.
[*Ar* : Mg, 24.3]

Reaction 1: $Mg(s) + H_2SO_4(aq) \rightarrow MgSO_4(aq) + H_2(g)$

amount of H_2SO_4 = 50/1000 x 1 = 0.05 mol

amount of Mg = 0.107 / 24.3 = 4.40 x 10^{-3} mol << 0.05

Mg is the limiting reagent.

$\Delta H = -\frac{q}{n_{Mg}} = -\frac{2016\ J}{4.40\times10^{-3}\ mol}$ = –458 kJ mol^{-1}

–458 kJ mol^{-1}

enthalpy change = ... [2]

(c) Determining the enthalpy change for Reaction 2

Read through the method before you start any practical work and prepare suitable tables for your results.

1. Weigh the stoppered tube containing **FA 4**. Record the mass in an appropriate format.
2. Support the plastic cup in the 250 cm^3 beaker.
3. Use a 50 cm^3 measuring cylinder to transfer 50.0 cm^3 of **FA 3** into the plastic cup.
4. Measure the temperature of **FA 3** in the plastic cup, using a 0.2 °C division thermometer and record the temperature.
5. Add the **FA 4** to the **FA 3** in the cup and stir the mixture constantly.
6. Measure and record the maximum temperature reached during the reaction.
7. Calculate and record the maximum temperature change that occurred during the reaction.
8. Weigh the stoppered tube that had contained **FA 4**. Record the mass.
9. Calculate and record the mass of **FA 4** added to the copper(II) sulfate.
10. Discard the contents of the plastic cup
11. Rinse the plastic cup and shake to dry.
12. Repeat this experiment using **FA 5** in place of **FA 4**.

Table 3: Reaction 2 - experiment 1

Mass of stoppered tube and **FA 4** /g	10.895
Mass of stoppered tube and residual **FA 4** /g	10.694
Mass of **FA 4** used /g	0.201
Initial temperature of **FA 3**/ °C	29.7
Maximum temperature reached/ °C	37.3
Change in temperature/ °C	+7.6

Table 4: Reaction 2 - experiment 2

Mass of stoppered tube and **FA 5** /g	10.764
Mass of stoppered tube and residual **FA 5** /g	10.567
Mass of **FA 5** used /g	0.197
Initial temperature of **FA 3**/ °C	29.6
Maximum temperature reached/ °C	36.8
Change in temperature/ °C	+7.2

(d) (i) Using data from **experiment 1**, show, using a suitable calculation, that the copper(II) sulfate was in excess in these reactions.

amount of $CuSO_4$ = 50/1000 x 1 = 0.05 mol

amount of Mg = 0.201 / 24.3 = 8.27 x 10^{-3} mol << 0.05

hence $CuSO_4$ is in excess.

[1]

(ii) Using your results, calculate the enthalpy change, in kJ mol^{-1}, for the reaction in experiment 1 and 2 respectively. Hence calculate the average enthalpy change, in kJ mol^{-1}, for the reaction between magnesium and copper(II) sulfate.
[Assume that 4.2 J is needed to raise the temperature of 1.0 cm^3 of solution by 1.0 °C.]
[*Ar* : Mg, 24.3]

For experiment 1,

$\Delta H_1 = -\frac{mc\Delta T}{n_{Mg}} = -\frac{50 \times 4.2 \times 7.6}{0.201/24.3} = -192.9$ kJ mol^{-1}

For experiment 2,

$\Delta H_2 = -\frac{mc\Delta T}{n_{Mg}} = -\frac{50 \times 4.2 \times 7.2}{0.197/24.3} = -186.5$ kJ mol^{-1}

$\Delta H_{average} = -190$ kJ mol^{-1}

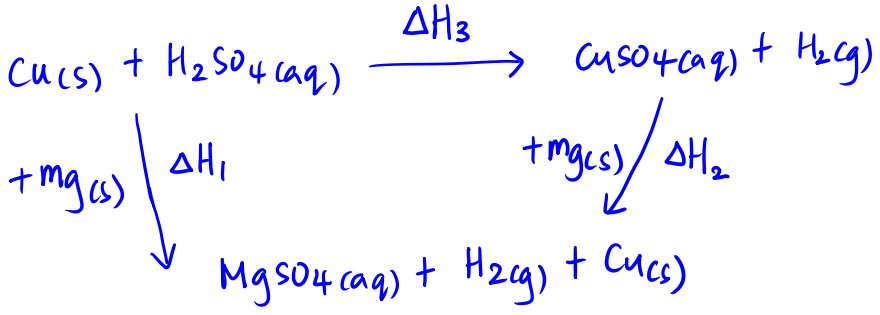
–190 kJ mol^{-1}
average enthalpy change = ... [3]

(e) **Reaction 3:** Cu(s) + H_2SO_4(aq) → $CuSO_4$(aq) + H_2(g)

Use your values for the enthalpy changes for **Reactions 1** and **2** to calculate the enthalpy change for **Reaction 3**.

Reaction 1: Mg(s) + H_2SO_4(aq) → $MgSO_4$(aq) + H_2(g)
Reaction 2: Mg(s) + $CuSO_4$(aq) → $MgSO_4$(aq) + Cu(s)

Show clearly how you obtained your answer.



Using Hess's law,
$\Delta H_{reaction\ 3} = \Delta H_{reaction\ 1} - \Delta H_{reaction\ 2} = -458 - (-190) = -268$ kJ mol^{-1}

–268 kJ mol^{-1}
enthalpy change = ... [1]

(f) **(i)** The method you used to determine the enthalpy change for **Reaction 1** was more accurate than the method you used to determine the enthalpy change for **Reaction 2**.

Suggest **two** reasons why the method used for **Reaction 2** was less accurate. Explain your answers.

Heat loss to surrounding is not accounted for in reaction 2, hence the magnitude of ΔT measured is less than actual and calculated $\Delta H_{\text{reaction 2}}$ is also less than actual.

ΔT of a lower magnitude is obtained for reaction 2 and hence there is higher percentage error due to measurement. ΔT measured and hence calculated $\Delta H_{\text{reaction 2}}$ is less accurate.

(ii) A student suggested that the accuracy of the method used for **Reaction 2** could be improved by using a larger volume of copper (II) sulfate. Explain if you agree with the student's suggestion. Give a reason for your answer.

No. Since copper (II) sulfate is in excess, the same amount of heat will be evolved. But having a larger volume of solution would result in a smaller magnitude of ΔT. Hence percentage error due to measurement would be higher.

OR

Yes. Since copper (II) sulfate is in excess, the same amount of heat will be evolved. But having a larger volume of solution would result in a smaller magnitude of ΔT. A smaller temperature rise would mean less heat would be lost to the surroundings.

[Data Processing] ; Thermochemistry Practical 3

3. Determination of ΔH_{rxn} of decomposition of H_2O_2

Hydrogen peroxide decomposes into water and oxygen. This is a slow spontaneous process that can be sped up using a catalyst such as manganese(IV) oxide.

$H_2O_2(aq) \rightarrow H_2O(l) + ½\ O_2(g)$

In this experiment, you are required to determine the enthalpy change, ΔH, for the catalytic decomposition of hydrogen peroxide.

You are given the following:

FA 1 is 0.10 mol dm^{-3} hydrogen peroxide, H_2O_2.
FA 2 is manganese(IV) oxide, MnO_2, the catalyst for the decomposition.

Procedure

Read through the method before starting any practical work and prepare a table for your results in the space below.

Experiment 1

1. Support the plastic cup inside a 250 cm^3 beaker.
2. Use a 50 cm^3 measuring cylinder to transfer 40.0 cm^3 of distilled water into the plastic cup.
3. Use another 50 cm^3 measuring cylinder to add 10.0 cm^3 of **FA 1** into the plastic cup.
4. Measure and record the initial temperature of the mixture using a 0.2 °C division thermometer.
5. Add a spatula measure of **FA 2** to the mixture in the plastic cup.
6. Stir constantly until the maximum temperature is reached and record this temperature.
7. Calculate and record the temperature rise.
8. Wash and wipe out your plastic cup and rinse the thermometer.

Experiment 2

Repeat steps 1 to 6 using 30.0 cm^3 of distilled water and 20.0 cm^3 of **FA 1.**

Experiment 3

Repeat steps 1 to 6 using 20.0 cm^3 of distilled water and 30.0 cm^3 of **FA 1.**

Experiment 4

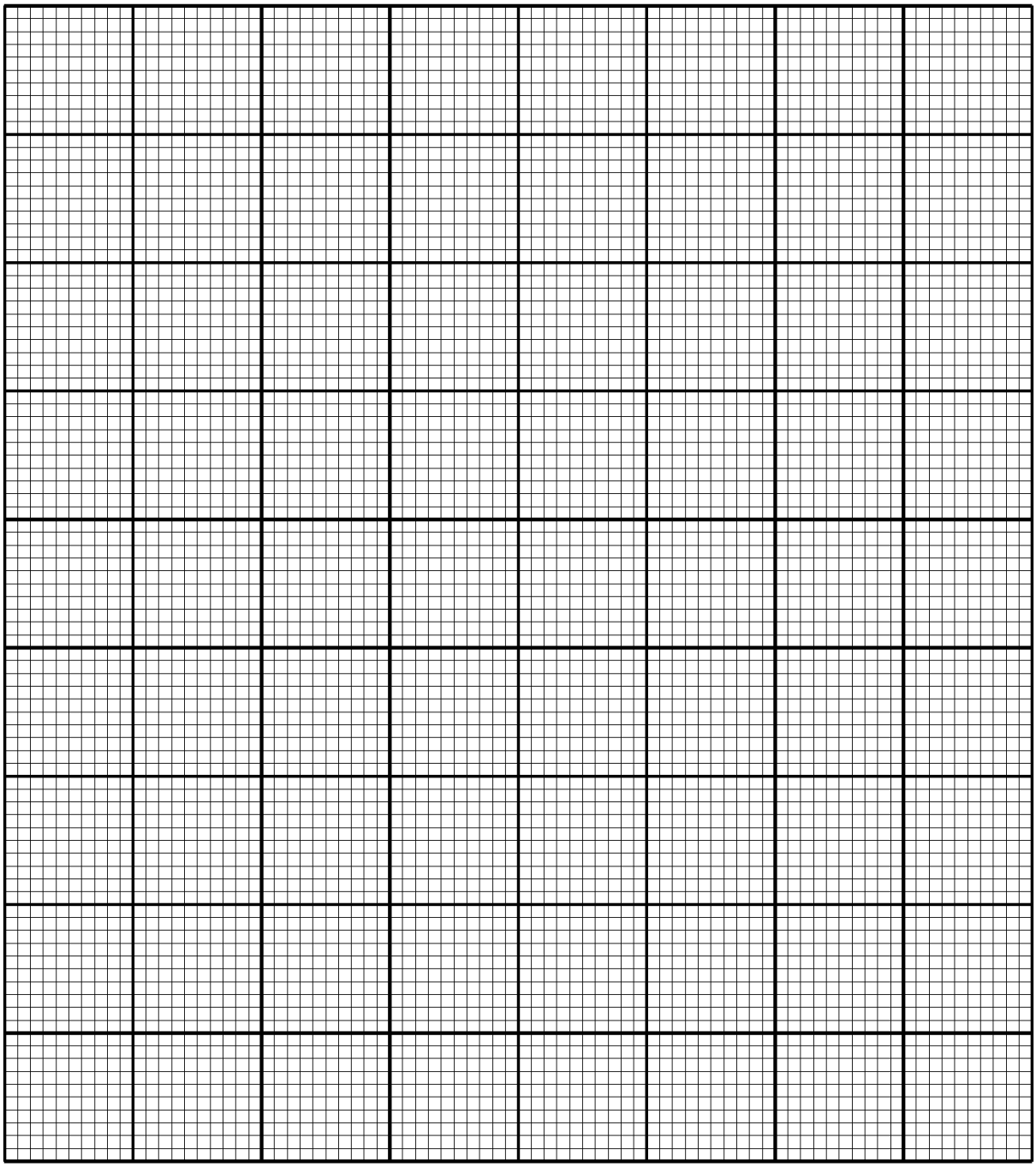
Repeat steps 1 to 6 using 10.0 cm^3 of distilled water and 40.0 cm^3 of **FA 1.**

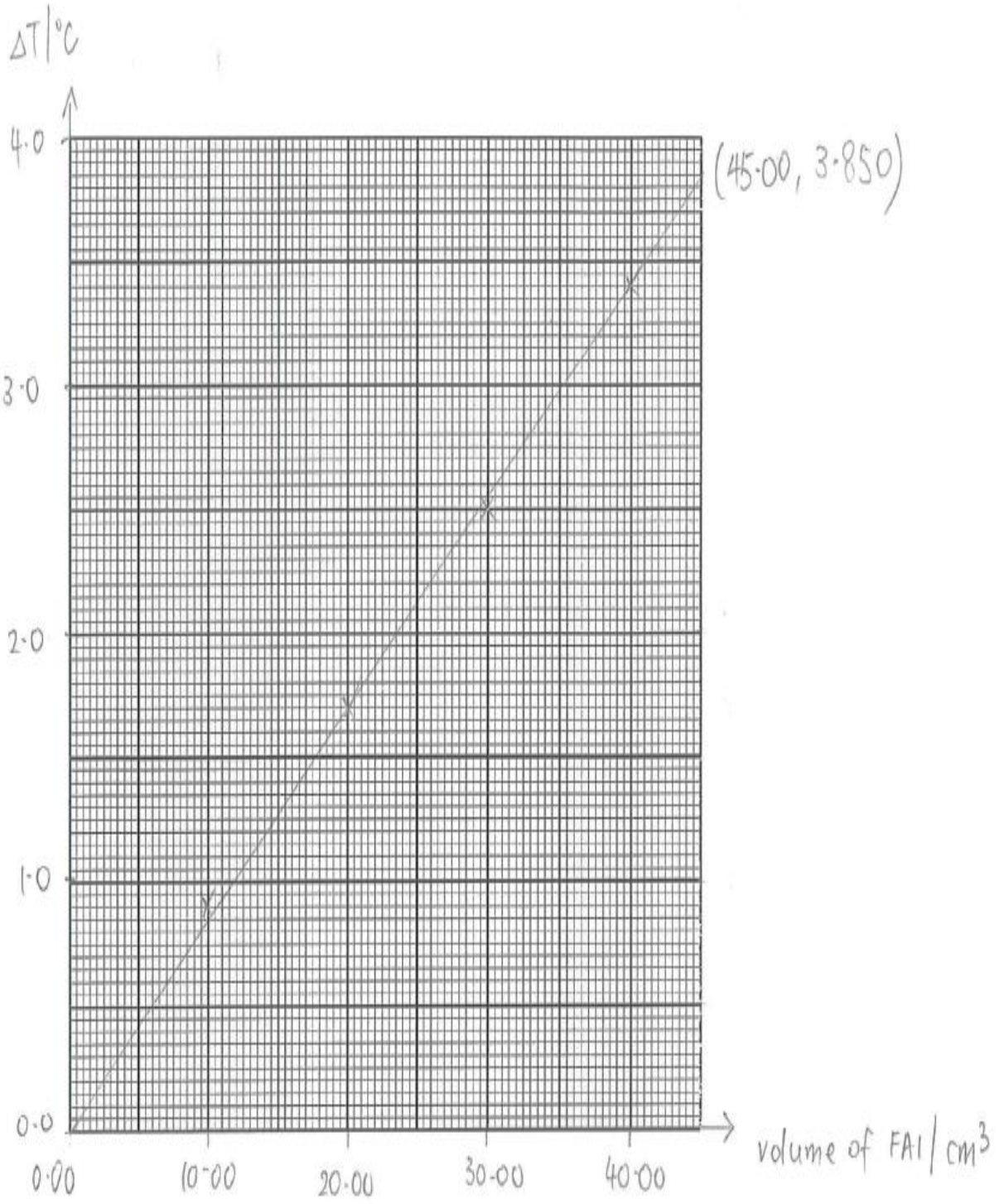
Results

Expt	Vol of FA 1/ cm^3	Vol of distilled water/ cm^3	Initial T/ ºC	Final T/ ºC	T rise/ ºC
1	10.0	40.0	30.1	31.0	0.9
2	20.0	30.0	30.1	31.8	1.7
3	30.0	20.0	30.1	32.6	2.5
4	40.0	10.0	30.1	33.5	3.4

(a) (i) Using the grid below, plot a graph of the temperature rise (*y*-axis) against the volume of **FA 1** (*x*-axis).

Draw the line of best fit.





[4]

(b) **(i)** Use your graph to extrapolate the temperature rise for 45.0 cm^3 of **FA 1** used. Show your working clearly on the graph.

+3.85 °C

temperature rise = ……. [1]

(ii) Calculate the energy released when 45.0 cm^3 of **FA 1** is used.
(Assume that 4.2 J are needed to raise the temperature of 1.0 cm^3 of solution by 1.0 °C.)

q = mcΔT = 50.0 x 4.2 x 3.850 = 808.5 = 809 J

Note: the total volume is kept constant at 50.0 cm^3.

809 J
energy released = ... [1]

(iii) Hence calculate the enthalpy change, in kJ mol^{-1}, for the reaction below.

$$H_2O_2(aq) \rightarrow H_2O(l) + 1/2\ O_2(g)$$

Amount of H_2O_2 = 0.1 x 45/1000 = 4.5 x 10^{-3} mol

$\Delta H = -\frac{q}{n_{H2O2}} = -\frac{808.5}{4.5 \times 10^{-3}}$ = –179667 = –180 kJ mol^{-1}

–180 kJ mol^{-1}
enthalpy change = ... [1]

(c) Which **one** of the four experiments that you carried out is likely to be the least accurate? Explain your choice.

Experiment 1 as the temperature rise is the smallest. Hence it has the highest percentage error due to measurement, making the data the least accurate.

(d) **(i)** Explain what the gradient of your graph represents.

The temperature rise per cm^3 of **FA 1** in the 50.0 cm^3 mixture.

(ii) Hence suggest the effect on the gradient of the graph if **FA 2** is not added in step **5**.

Without **FA 2** to act as a catalyst, the reaction will be slower. There is more heat loss to surrounding, resulting the ΔT per cm^3 of **FA 1** to be lower. Hence gradient will be smaller / less steep.

[Data Processing] ; Thermochemistry Practical 4

4. Determination of ΔH_{sol} of NH_4Cl

When an exothermic reaction takes place in a container such as a beaker, some of the evolved heat energy is absorbed by the beaker.

When an endothermic reaction takes place some of the required heat energy is supplied by the beaker.

The amount of heat energy evolved or supplied for a 1°C change in temperature is known as the heat capacity of the beaker.

In this experiment, three reactions are involved.

NH_4Cl (s) + NaOH (aq) → NH_3 (aq) + $NaCl$ (aq) + H_2O (l) $\quad \Delta H_{reaction1}$
NH_4Cl (s) + aq → NH_4Cl (aq) $\quad \Delta H_{solution} = +14$ kJmol^{-1}
NH_4Cl (aq) + NaOH(aq) → NH_3 (aq) + $NaCl$ (aq) + H_2O(l) $\quad \Delta H_{reaction2}$

This experiment requires you to determine the following

- The heat capacity of the calorimeter.
- $\Delta H_{reaction1}$

$\Delta H_{reaction2}$ will be determined using Hess' law.

You are given the following:

FA 1 is NH_4Cl solid
FA 2 is 2.00 mol dm^{-3} sodium hydroxide, NaOH.

(a) Determining the approximate heat capacity of the 250 cm^3 beaker

When samples of hot and cold water are mixed in the 250 cm^3 beaker, some heat is lost to the beaker in raising its temperature. To determine the approximate heat capacity of your 250 cm^3 beaker, you will determine the maximum temperature rise when a sample of hot water is added to tap water in the beaker.

1. Use a 50 cm^3 measuring cylinder to transfer 50.0 cm^3 of tap water into the 250 cm^3 beaker.
2. Use the 50 cm^3 measuring cylinder to transfer 50.0 cm^3 of tap water into a 100 cm^3 beaker. Note the temperature of the water in this 100 cm^3 beaker.
3. Heat the content of 100 cm^3 beaker **carefully and gently** until the temperature of the water has increased by 45–50 °C then stop heating. *e.g. if the water is at 20.0 °C, you should warm it to 65–70 °C*.
4. Stir the tap water in the 250 cm^3 beaker with the thermometer.
5. Record the temperature of the tap water (this is the temperature at t = 0 min).
6. Record the temperature each minute for 3 minutes.
7. After you have taken the reading at t = 3 min, use the thermometer to stir the hot water in the 100 cm^3 beaker.
8. At t = 4 min, measure the temperature of the hot water and record this value in the blank below.

9. **Immediately** add the hot water from the 100 cm^3 beaker to the tap water in the 250 cm^3 beaker. Stir with the thermometer but do **not** record the temperature.
10. Continue to stir the water throughout the experiment.
11. Record the temperature at t = 5 min, and then every **½ minute** until t = 8 min.
12. Empty and rinse the 250 cm^3 beaker. Dry it using a paper towel.
13. Record all measurements of time and temperature obtained.

The temperature, T_1, of the hot water at t = 4 min is 95.0 °C

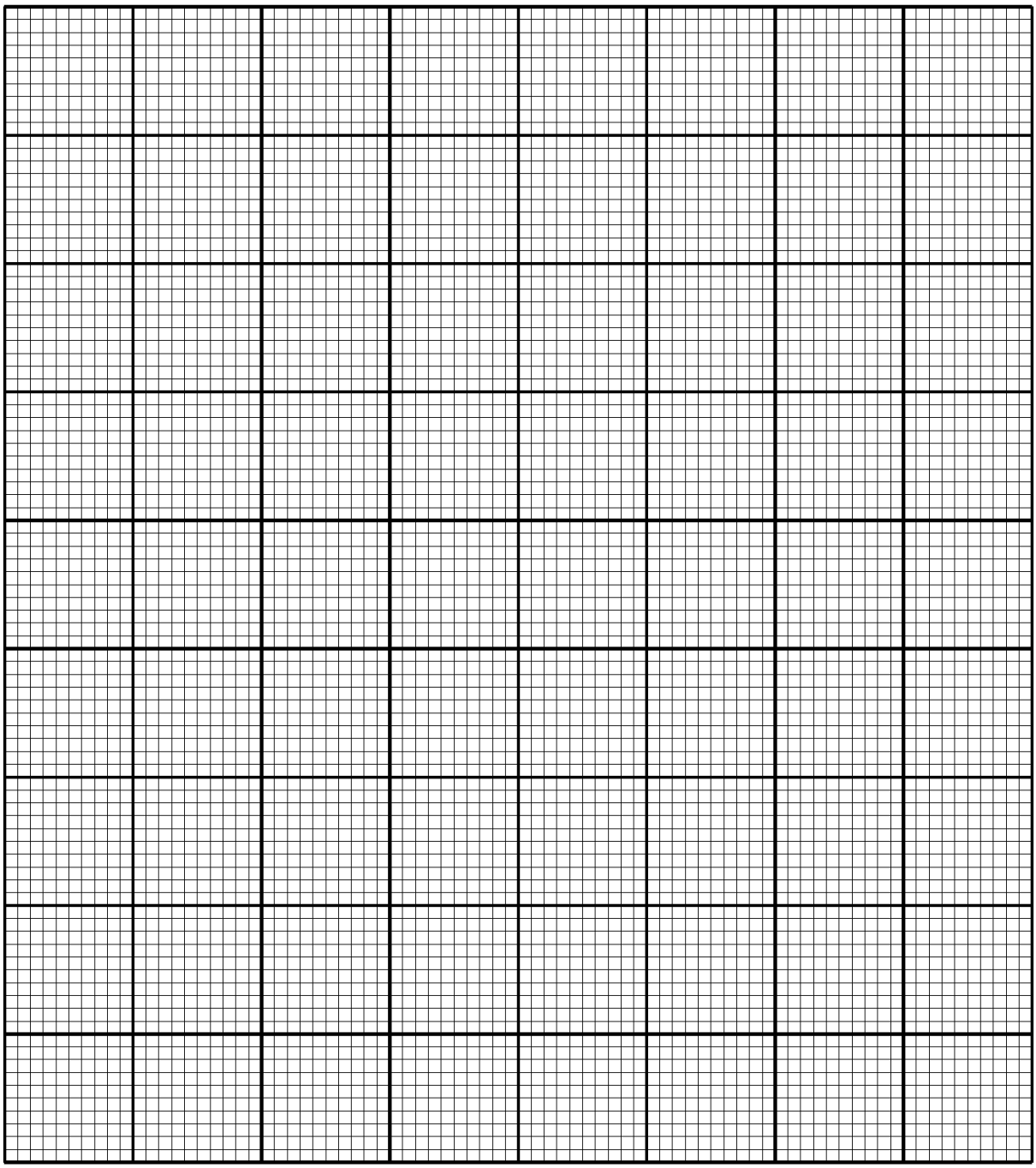
Results

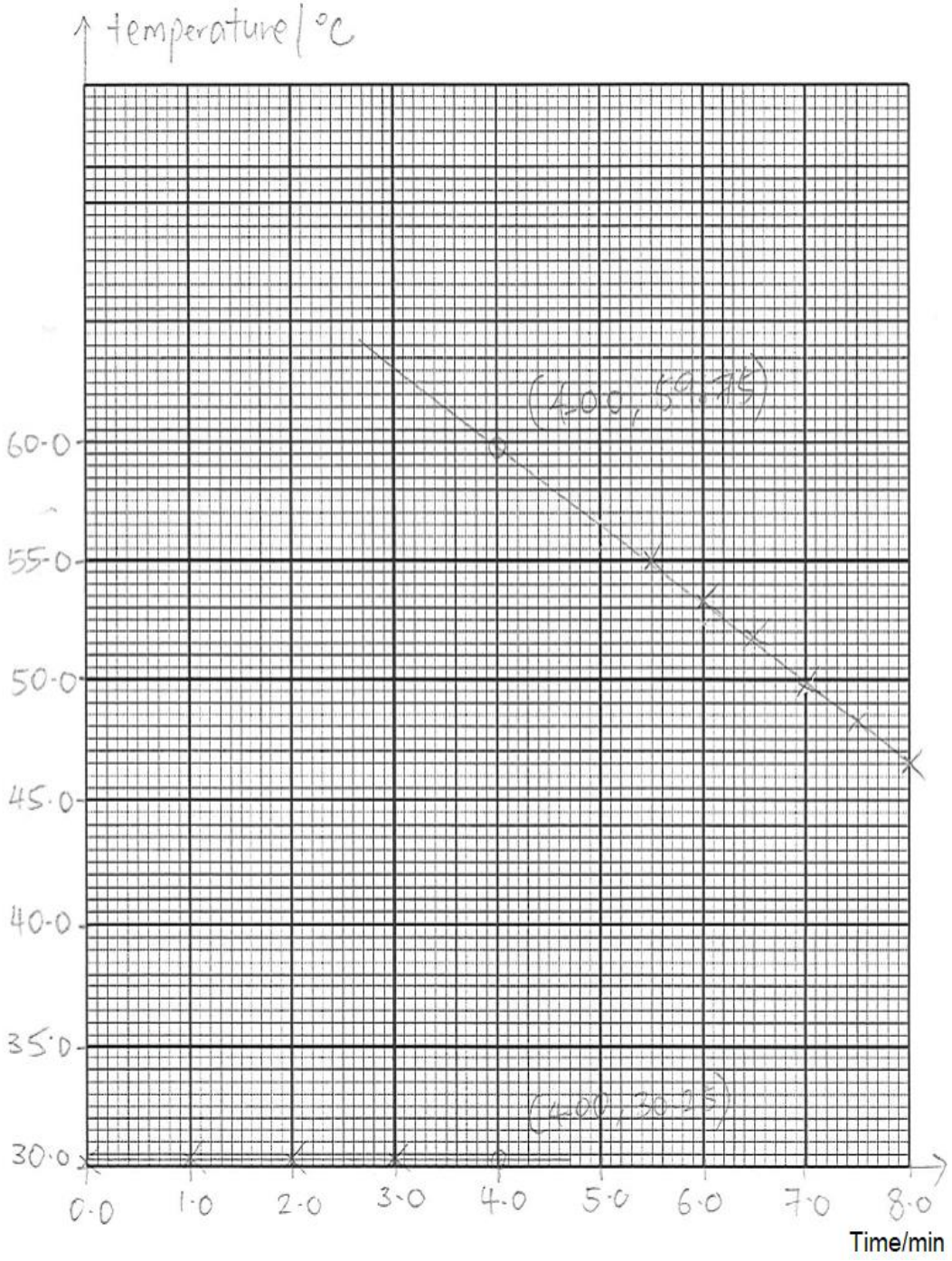
Temperature of tap water in 100 cm^3 beaker : 30.2 °C

Time/ min	Temperature/ °C	Time/ min	Temperature/ °C
0.0	30.2	**5.5**	55.0
1.0	30.2	**6.0**	53.2
2.0	30.2	**6.5**	51.6
3.0	30.2	**7.0**	49.8
4.0	-	**7.5**	48.2
5.0	56.6	**8.0**	46.5

(b) Graph plotting

1. Plot a graph of the temperature of the water in the 250 cm^3 beaker (y-axis) against time (x-axis) on the grid below. Do **not** plot the temperature, T_1, of the hot water at t = 4 min.
2. Draw two straight lines of best fit; one through the points up to t = 3 min; the second through the points from t = 5 min to t = 8 min. Extrapolate both lines to t = 4 min.
3. From the extrapolated lines read the minimum and the maximum temperatures at t = 4 min. Record these values in the spaces provided below.
4. Determine the values for the two temperature changes at t = 4 min.





Minimum temperature, T_2, at t = 4 min is 30.25 °C.

Maximum temperature, T_3, at t = 4 min is 59.75 °C.

Temperature rise for 50.0 cm^3 of tap water in the 250 cm^3 beaker, ($T_3 - T_2$) is 29.50 °C.

Temperature fall for 50.0 cm^3 of hot water from the 100 cm^3 beaker, ($T_1 - T_3$)
35.25
is ..... °C.

(c) **(i)** Calculate the heat energy gained by the 50.0 cm^3 of tap water in the 250 cm^3 beaker.
[**4.2 J** are absorbed or released when the temperature of 1.0 cm^3 of **water** changes by 1.0 °C.]

q = 50.0 x 4.2 x 29.50 = 6195 = 6200 J

6200 J
heat gained = ... [1]

(ii) Calculate the heat energy lost by the 50.0 cm^3 of hot water from the 100 cm^3 beaker.

q = 50.0 x 4.2 x 35.25 = 7402.5 = 7400 J

7400 J
heat lost = ... [1]

(iii) The difference between the values calculated in **(i)** and **(ii)** is an approximate value for the total heat energy absorbed by the 250 cm^3 beaker during the experiment.

The heat capacity of the beaker is the amount of heat energy absorbed for a 1 °C change in temperature.

approximate heat capacity of the 250 cm^3 beaker

$$= \frac{(\text{heat energy lost}) - (\text{heat energy gained})}{T_3 - T_2} \text{ J °C}^{-1}$$

Use your answers to **(i)** and **(ii)** and the temperature rise from **(b)** to calculate the approximate heat capacity of the 250 cm^3 beaker.

Approximate heat capacity = (7402.5 – 6195) ÷ (29.50) = 40.9 J $°C^{-1}$

40.9 J $°C^{-1}$
heat capacity of the 250 cm^3 beaker = ... [1]

(d) Determining the enthalpy change of solution for ammonium chloride

Record all your mass and temperature measurements in the table below.

1. Weigh the stoppered tube containing ammonium chloride, which is labelled **NH_4Cl**.
2. Use the 100 cm^3 measuring cylinder to transfer 100.0 cm^3 of tap water into the rinsed and dried 250 cm^3 beaker used in **(a)**.
3. Stir the water in the beaker with the thermometer and record the temperature.
4. Add the solid from the weighed tube to the water. Stir the mixture constantly with the thermometer.
5. Record the minimum temperature obtained in the solution.
6. Reweigh the tube labelled **NH_4Cl**, its stopper and any residual ammonium chloride.
7. Empty and rinse the beaker and dry it using a paper towel.

Results

mass of tube + ammonium chloride / g	10.007
mass of tube + residual ammonium chloride / g	5.012
mass of ammonium chloride / g	4.995
initial temperature of water / °C	30.1
minimum temperature obtained / °C	27.7
temperature fall, **ΔT** / °C	− 2.4

(e) (i) Use the temperature fall from the experiment, to calculate the change in heat energy of the solution.
[**4.2 J** are absorbed or released when the temperature of 1.0 cm^3 of **solution** changes by 1.0 °C.]

$q = mc\Delta T = 100.0 \times 4.2 \times (-2.4) = 1008 = -1010$ J

−1010 J
heat change = …….. [1]

(ii) To calculate the total change in heat energy as ammonium chloride dissolves in water, the change in heat energy of the 250 cm^3 beaker has to be added to the change in heat energy of the solution.

Explain why these two changes in heat energy have to be added together.

Heat required for the endothermic reaction of the dissolution of ammonium chloride is equivalent to the heat absorbed from the surrounding, which includes both the solution and beaker.

(iii) Use your answer in **(i)** above and the approximate heat capacity of the 250 cm^3 beaker calculated in **(c)(iii)** to calculate the combined change in heat energy of the beaker and solution.

$q_{combine} = q_{beaker} + q$
$= (-2.4)(40.9) - 1008 = -1106 = -1110$ J

change in heat energy = – 1110 J [1]

(iv) Calculate how many moles of **FA 1**, NH_4Cl, were used in the experiment.
[*Ar*; C*l*, 35.5; H, 1.0; N, 14.0]

Amount of NH_4Cl = 4.995 / 53.5 = 0.0934 mol

moles of **FA 1** = 0.0934 mol [1]

(v) Calculate the enthalpy change of solution, $\Delta H_{solution}$ (NH_4Cl).

$\Delta H_{solution}$ (NH_4Cl) = $-\frac{q}{0.0934} = -\frac{-(1106\ J)}{0.0934\ mol}$ = +11.8 kJ mol^{-1}

$\Delta H_{solution}$ (NH_4Cl) = +11.8 kJ mol^{-1} [1]

(vi) Explain the significance of the sign you have given in **(v)** and how it is related to your experimental results.

The positive sign shows that the reaction is endothermic (heat is absorbed during the reaction) as the products have higher energy content than the reactants. Since heat energy is absorbed from the surroundings for the reaction, the temperature of the solution falls.

(f) **(i)** A data book value for the molar enthalpy change of solution, $\Delta H_{solution}$ (NH_4Cl), is +15.2 kJ mol^{-1}. The value you have obtained may be significantly different from this value.

Calculate the difference between your value of $\Delta H_{solution}$ (NH_4Cl) and the data book value. Record this difference below. Express this difference as a percentage of the data book value.

Difference = +15.2 – 11.8 = –3.4 kJ mol^{-1}
% difference = –3.4 / 15.2 x 100 % = –22.4 %

percentage difference = –22.4 % [1]

(ii) Describe **one** major source of error in this experiment. Suggest an improvement which would significantly increase the accuracy of the experiment. Explain why your suggestion would produce a more accurate value.

A small magnitude of ΔT is measured, which results in high percentage error due to measurement. Use a larger mass of NH_4Cl (double to 10 g) or smaller volume of water (reduce to 25.0 cm^3) to obtain a larger magnitude of ΔT, hence lowering the percentage error in measurement.

Use of glass beaker as calorimeter, which has a higher heat capacity than Styrofoam cup. This results in a lower magnitude of ΔT, hence magnitude of calculated ΔH will be less than actual. Change to use Styrofoam cup with a lower heat capacity as the calorimeter. ΔT measured will be closer to actual.

Data Processing

[Data Processing] : *Kinetics Practical 1*

1. Determination of the relationship between rate of decomposition of H_2O_2 and its concentration.

Two experiments on the decomposition of H_2O_2 in the presence of a catalyst, I^- will be carried out.

The reactions involved are:

$$H_2O_2\ (aq) + I^-\ (aq) \rightarrow IO^-\ (aq) + H_2O\ (l)$$
$$2IO^-\ (aq) \rightarrow 2I^-\ (aq) + O_2\ (g)$$

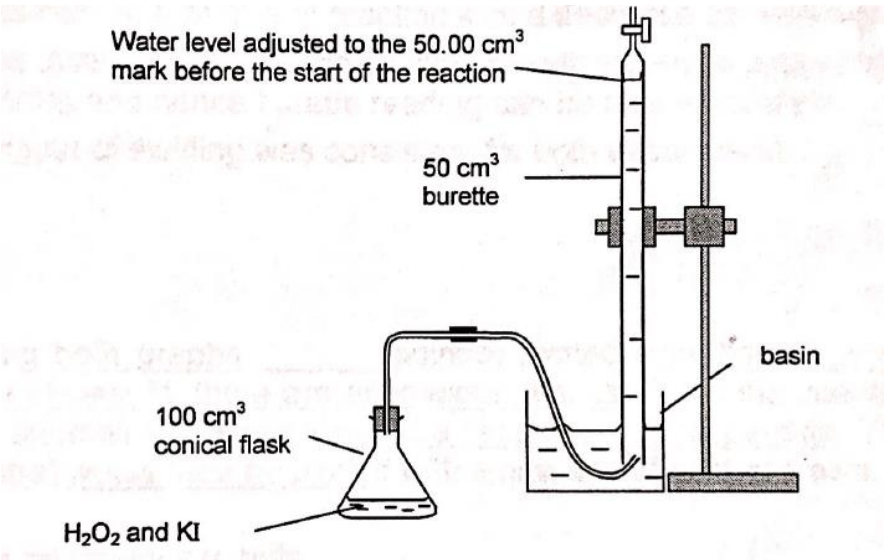
Overall: $2H_2O_2\ (aq) \rightarrow 2H_2O\ (l) + O_2\ (g)$

This reaction can be followed by measuring the volume of oxygen gas evolved as the reaction proceeds.

You are provided with the following:
Aqueous hydrogen peroxide
Aqueous potassium iodide

Procedure:

1. Fill a burette with water. By holding a thumb tightly over the mouth of the burette, invert it over a beaker of water.
2. By controlling the tap of the burette, slowly allow some air to enter until water level corresponds to the 50.00 cm^3 mark. (or until you are able to read off a volume from the burette)
3. Record the initial burette reading.
4. Place the rubber tubing such that the gas evolved will be collected in the burette via downward displacement of water as shown in the diagram.



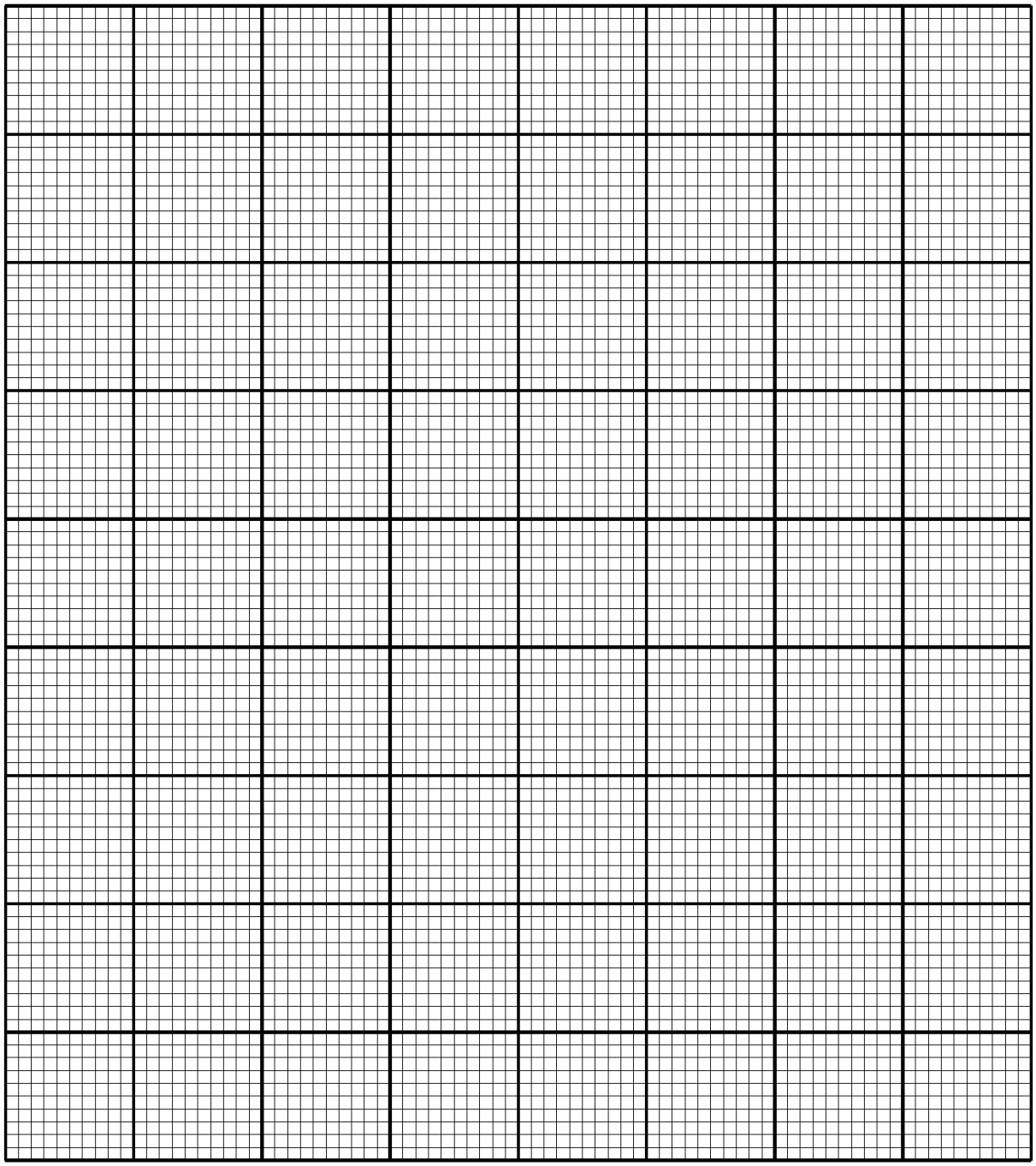
5. Pipette 20.0 cm^3 of hydrogen peroxide into the conical flask.
6. Measure 10 cm^3 of potassium iodide in a measuring cylinder.
7. Remove the stopper from the conical flask. Start the stopwatch the instant when potassium iodide is added to the conical flask and immediately replace the stopper.
8. Swirl the conical flask once immediately to ensure even mixing.
9. Do not swirl the conical flask throughout the experiment.
10. Record the burette reading every 30 seconds for the first 3 minutes and continue to record for every 1 minute for a total duration of 8 minutes.
11. For the second experiment, rinse out the conical flask and add 10 cm^3 of distilled water into a second measuring cylinder.
12. Pipette 10.0 cm^3 of hydrogen peroxide into the conical flask and add 10 cm^3 of distilled water from the second measuring cylinder.
13. Repeat steps 6 to 9.

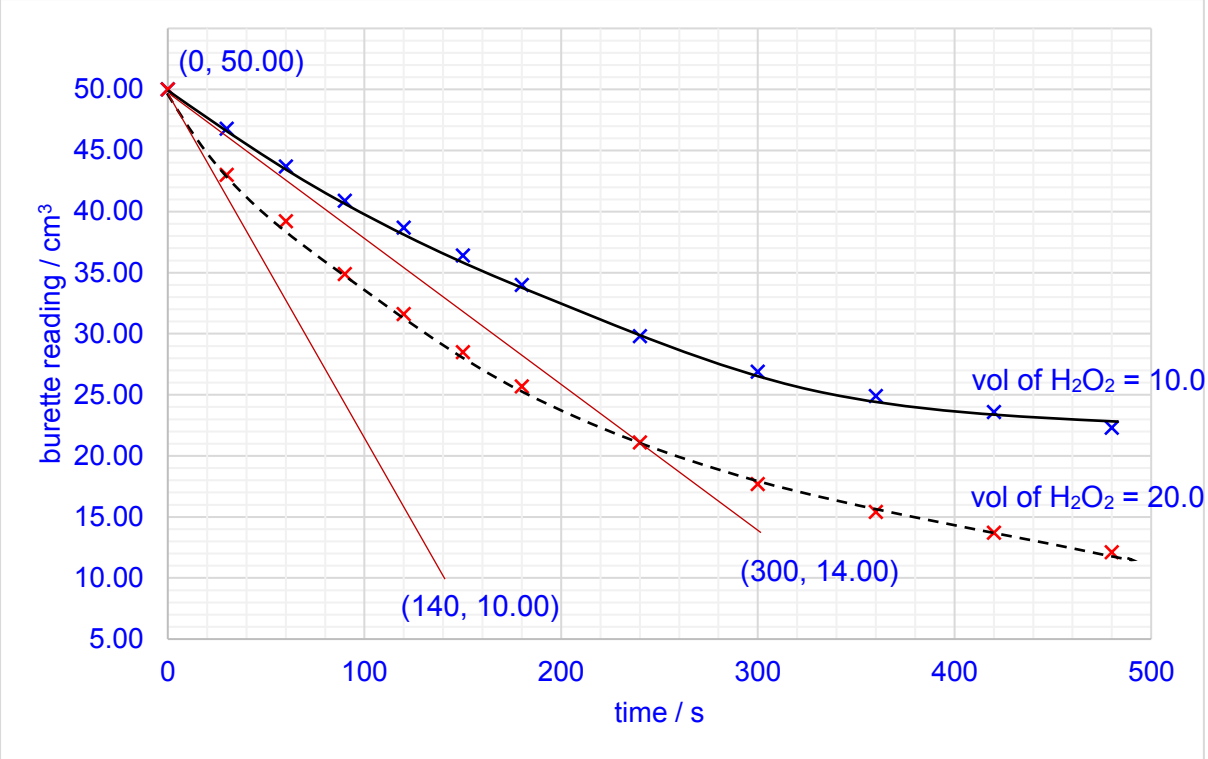
Results

Time/s	Expt 2 10.0 cm^3 H_2O_2	Expt 1 20.0 cm^3 H_2O_2
	Burette reading/ cm^3	Burette reading/ cm^3
0	50.00	50.00
30	46.80	43.00
60	43.70	39.20
90	40.90	34.90
120	38.70	31.60
150	36.40	28.50
180	34.00	25.70
240	29.80	21.10
300	26.90	17.70
360	24.90	15.40
420	23.60	13.70
480	22.30	12.10

(a) For each of the experiment, plot a graph of burette reading against time on the same graph grid provided.

The rate of this reaction can be investigated by analysing the graph of burette reading against time.





[4]

(i) Using the graph plotted, calculate the initial relative rate of the two graphs. Show your working clearly.

For 10.00 cm^3 of H_2O_2,

relative rate = $\frac{50.00-14.00}{300}$ = 0.120 $cm^3\ s^{-1}$

For 20.00 cm^3 of H_2O_2,

relative rate = $\frac{50.00-10.00}{140}$ = 0.286 $cm^3\ s^{-1}$

(ii) State and explain the relationship between the volume of H_2O_2 (aq), V_{H2O2} and the initial concentration of H_2O_2, $[H_2O_2]_{mixture}$ in the reaction mixture.

The initial concentration of H_2O_2 is **directly proportional** to the volume of hydrogen peroxide used.

$$[H_2O_2]_{mixture} = \frac{\frac{V_{H2O2}}{1000} \times [H_2O_2]_{original}}{\text{total vol of rxn mixture}/_{1000}}$$

$$= \frac{V_{H2O2} \times [H_2O_2]_{original}}{\text{total vol of rxn mixture}}$$

$[H_2O_2]_{original}$ and the total volume of the reaction mixture is kept constant at 30 cm^3, thus $[H_2O_2]_{mixture} \propto V_{H2O2}$ added to the mixture.

(iii) By comparing the gradient of the two graphs that correspond to different initial concentrations of H_2O_2, deduce the order of the reaction with respect to H_2O_2.

The relative rate approximately doubles from 0.120 to 0.289 $cm^3\ s^{-1}$ when the concentration doubles (i.e. volume doubles from 10 to 20 cm^3), the order with respect to hydrogen peroxide is 1.

order with respect to H_2O_2 = 1 ..... [2]

(iv) Suggest how you can carry out a third experiment to determine the order of reaction with respect to I^-.

With reference to experiment **2**, react 20.0 cm^3 of I^- with 10.0 cm^3 of H_2O_2. (must keep total volume of reaction mixture constant at 30 cm^3)

(b) Explain the importance of step **8** in the procedure.

The swirling of the mixture at the start is to ensure that the reaction mixture is ***homogeneous*** so that the rate is uniform across the entire reaction mixture.

Hence the order of reaction with respect to the hydrogen peroxide can be determined accurately.

[Data Processing] ***: Kinetics Practical 2***

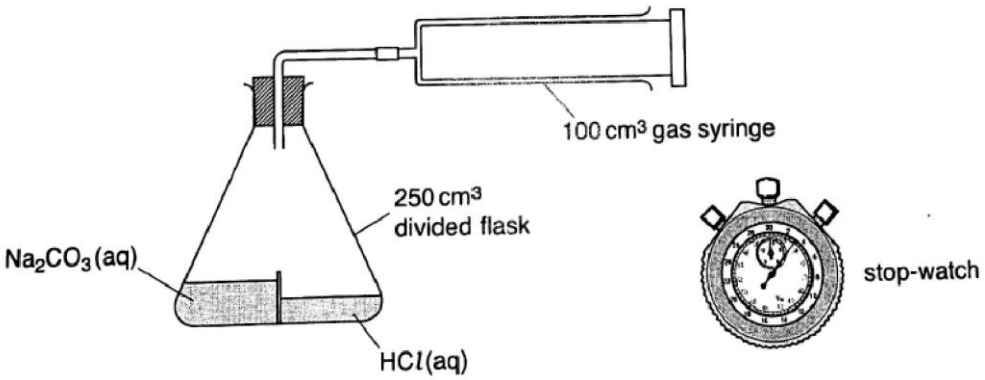
The kinetics of the reaction between Na_2CO_3 and HC*l* is studied. The order of reaction with respect to HC*l* can be investigated using the following set-up by varying the concentration of HC*l*.

Method

(a) Results

1. 100.0 cm^3 of 4 mol dm^{-3} Na_2CO_3 is pipetted into one side of the divided flask.
   Amt = 0.4 mol
2. 20.0 cm^3 of 5.0 mol dm^{-3} HC*l* is pipetted into the other side of the divided flask.
   Amt = 0.10 mol (limiting agent) 100 % yield = 01 x 24000cm^3, 20 cm^3 is 20/2400 = 1/120 of 100% yield.
3. The apparatus is assembled as shown and the reaction started by swirling the flask to mix the solutions.

.



4. The stop watch is started at the moment the reaction commences.
5. The stop watch is stopped and the time recorded when 20 cm^3 of gas have been collected.
6. The apparatus is rinsed out and the experiment repeated using volumes of acid and water shown in **Table 1**.

The acid and water volumes are measured using a graduated pipette.

[3]

Results:

Complete the following table using the following expressions:

- **Initial rate of reaction =** $\frac{20}{time\ taken\ for\ gas\ collection}$

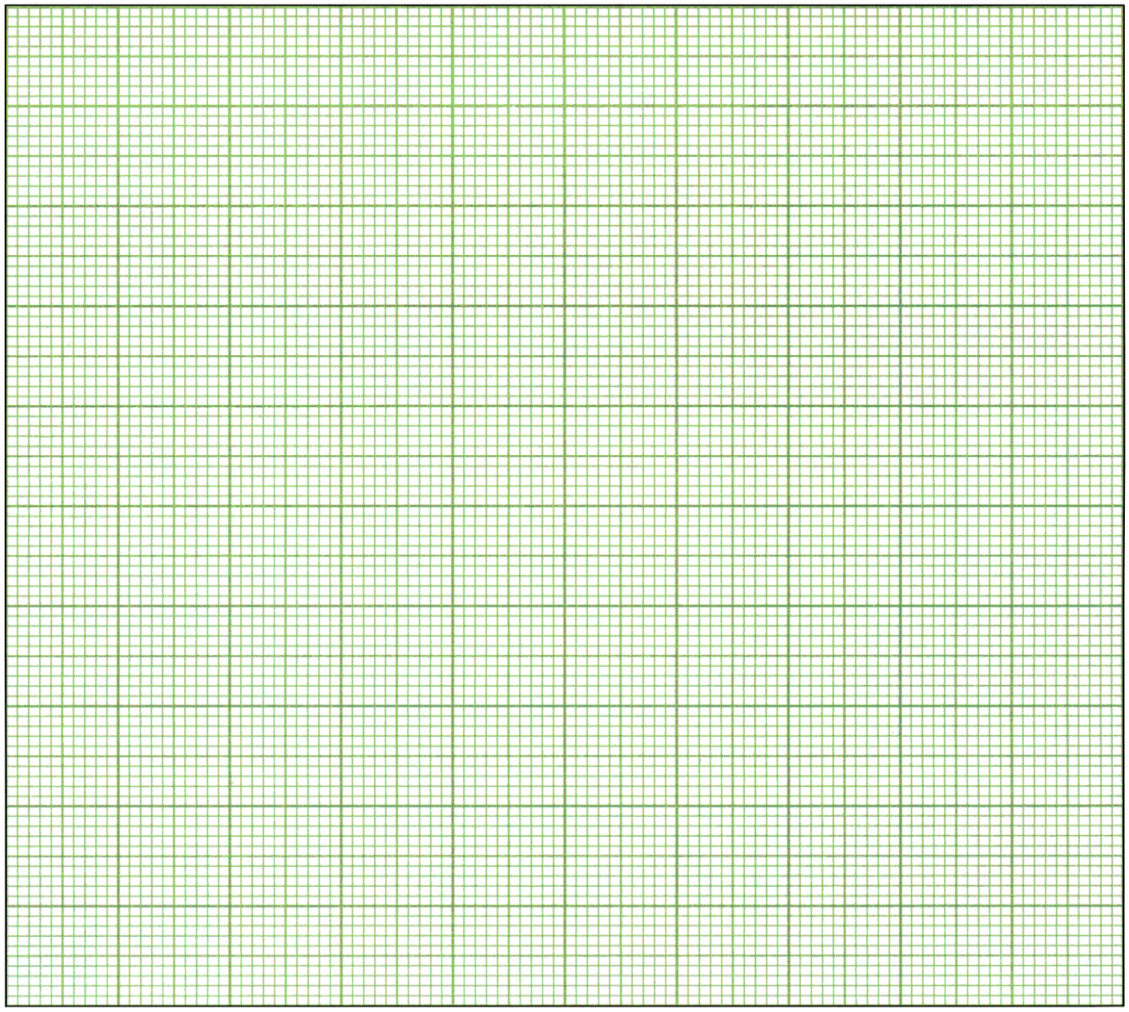
Relative [HC*l*] = $\frac{volume\ of\ acid\ in\ numbered\ experiment}{volume\ of\ acid\ in\ experiment\ 1}$

Record all values of initial rate of reaction and relative [HC*l*] to 2 significant figures.

Expt	Volume of 5.0 mol dm^{-3} HC*l* added / cm^3	Volume of water added / cm^3	Volume of gas collected / cm^3	Time taken to collect 20 cm^3 gas / s	Initial rate of reaction / $cm^3\ s^{-1}$	Relative [HC*l*]
1	20.0	0	20	8	***2.50***	***1.0***
2	17.0	3.0	20	9	***2.22***	***0.85***
3	14.0	6.0	20	12	***1.67***	***0.70***
4	11.0	9.0	20	14	***1.43***	***0.55***
5	8.0	12.0	20	20	***1.00***	***0.40***
6	5.0	15.0	20	32	***0.63***	***0.25***
7	2.0	18.0	20	80	***0.25***	***0.10***

Table 1

(b) Plot the initial rate of reaction against the relative concentration of the acid. Start each scale at zero. Draw the line of best-fit through the points.



(c) What can you conclude from your plot about the order of reaction with respect to hydrochloric acid? Explain your answer.

The graph is a straight line that passes through the origin, showing the rate of reaction is directly proportional to the concentration of HC*l*.

Therefore the order of reaction wrt HC*l* is 1.

(d) A student carrying out the experiment made a mistake and only pipetted 50 cm^3 rather than 100 cm^3 of 0.4 mol dm^{-3} sodium carbonate into the flask.
How will this affect the rate of reaction? Explain your answer.

The total volume of reaction mixture has decreased from the previous 120 cm^3 to 70 cm^3, hence the concentration of HC*l* has increased. The rate of reaction will therefore increase. The time taken to produce 20 cm^3 of CO_2 will be shorter as the rate of reaction is faster than previous.

2 Alternative manipulation of experimental results to determine order of reaction with respect to HC*l*.

Calculate the values of (volume of **HC*l*** × reaction time) in the table below.

Expt	Volume of 5.0 mol dm^{-3} HC*l* added / cm^3	Volume of water added / cm^3	Volume of gas collected / cm^3	Time taken to collect 20 cm^3 gas / s	vol $_{HCl}$ x t / cm^3 s
1	20.0	0	20	8	***160***
2	17.0	3.0	20	9	***153***
3	14.0	6.0	20	12	***168***
4	11.0	9.0	20	14	***154***
5	8.0	12.0	20	20	***160***
6	5.0	15.0	20	32	***160***
7	2.0	18.0	20	80	***160***

(a) Explain how the values of (vol$_{HCl}$ × t) of these experiments show that the order of reaction with respect to HC*l* is in agreement to your answer determined in **(c)** on pg 50**.**

vol $_{HCl}$ × t for all the experiments is relatively constant / ≈ 160 cm^3 s

vol $_{HCl}$ × t = constant

vol $_{HCl} \propto \frac{1}{t}$ (in this experiment, vol $_{HCl} \propto$ [HC*l*] while $\frac{1}{t} \propto$ rate)

hence rate α [HC*l*], which means that order of reaction wrt HC*l* is 1 and this is in agreement with the analysis done in part (c).

*Note to Teacher: Ask students to consider the relationship between v and t for a 2nd order reaction.

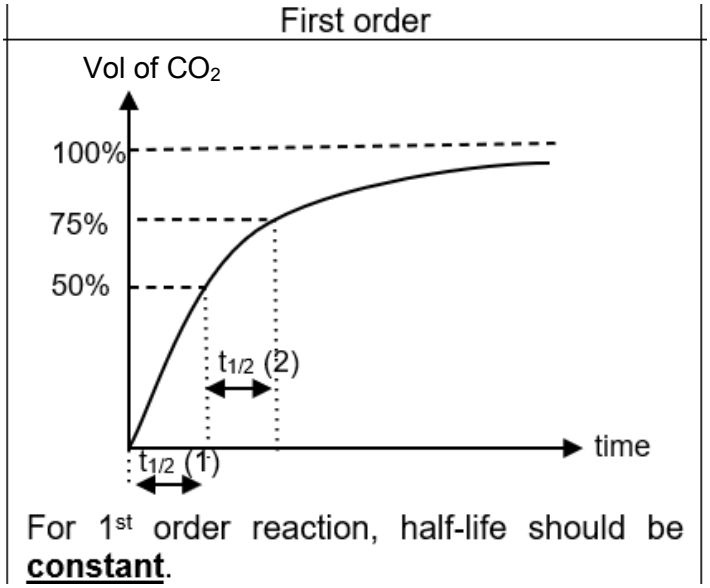
v^2t = constant

If t is constant for different v, order = 0

(b) The 1st order of reaction with respect to HC*l* in this reaction can also be verified to by monitoring the volume of carbon dioxide gas during the course of reaction in experiment **7** of table 1.

In the space below, sketch the graph you would expect to obtain when the reaction is allowed to proceed to completion. You may assume that the total volume of carbon dioxide gas collected in the gas syringe is $\mathbf{V_{max}}$.

Explain your answer.



Explanation

With $[Na_2CO_3]$ in large excess and thus effectively constant during the course of reaction, rate of this reaction depends on $[H^+]$ only.
Since order of reaction wrt HC*l* is 1, $t_{½}$ is constant (i.e. $t_{½(1)} \approx t_{½(2)}$)

[Data Processing] ***: Kinetics Practical 3***

2. To investigate the reaction between substance X, iodine and hydrogen ions.

You are provided with the following:

FA 4 is 1.00 mol dm^{-3} sulfuric acid
FA 5 is an aqueous solution of substance **X**
FA 6 is 0.00375 mol dm^{-3} iodine, I_2

Procedure:

1. Using a measuring cylinder, measure out 20 cm^3 of **FA 4** and 20 cm^3 of **FA 5**, as shown in column 1 of **Table 1**, into a conical flask. It is not necessary to rinse the measuring cylinder between solutions.
2. Measure out 4.00 cm^3 of **FA 6** from the burette into a test-tube.
3. Start the reaction by tipping the **FA 6** from the test-tube into the conical flask. Start the stop-clock with one hand and swirl the contents of the flask with the other. Place the flask on a white tile and stop the clock as soon as the yellow colour disappears.
4. Record the time in seconds (to the nearest second) in **Table 1**.
5. Repeat the experiment using the different volumes of **FA 4**, **FA 5** and **FA 6** as shown in **Table 1**. Where water is required, use the measuring cylinder to add the water to the other solutions in the conical flask.

The "rate of reaction" can be calculated by using the relationship:

$$\text{rate} \propto \frac{\text{volume of FA 6 in cm}^3}{\text{time in seconds for colour to disappear}}$$

(a) Results

Calculate each rate and complete **Table 1**.

Table 1

Experiment	**1**	**2**	**3**	**4**
Volume of **FA 4** /cm^3	20.0	20.0	10.0	20.0
Volume of **FA 5** /cm^3	20.0	20.0	20.0	10.0
Volume of water /cm^3	0.0	2.0	10.0	10.0
Volume of **FA 6** /cm^3	4.0	2.0	4.0	4.0
Time for colour to disappear /s	24	24	48	48
"rate of reaction"	0.167	0.0833	0.0833	0.0833

[3]

(b) Explain the importance of adding 10 cm^3 of deionized water for **experiment 3** and **4** and the effect it would have on the dependent variable if not adhered to.

Adding 10 cm^3 of deionized water ensures that ***the total volume of reaction mixture is kept constant*** in both experiments. Hence, the ***volume of reagent added is directly proportional to its concentration*** in the reaction mixture.

If this step is not done, the concentration of all reagents are higher than expected, leading to a faster rate. Hence, time taken for the colour to disappear will be ***lower*** than expected.
Concentration of each reactant in each reaction mixture has to be calculated so that concentration of reactants can be compared between experiments.

(c) **(i)** From **Table 1**, determine the order of reaction with respect to

- [**X**]

compare expt 1 and 4,
when [**X**] halves (volume of FA 5 halved), the rate also halved from 0.166 to 0.0833, this suggests that order of reaction with respect to **X** is 1.

order of reaction with respect to [**X**] = 1 ……

- $[H^+]$

compare expt 1 and 3,
when $[H^+]$ halves (volume of FA 4 halves), the rate also halved from 0.166 to 0.0833, this suggests that order of reaction with respect to H^+ is 1.

order of reaction with respect to $[H^+]$ = 1 ……

- $[I_2]$

compare expt 1 and 2,
when $[I_2]$ halves (volume of FA 6 halves), the rate also halves from 0.166 to 0.0833, this suggests that order of reaction with respect to I_2 is 1.

order of reaction with respect to $[I_2]$ = 1 ……

(ii) Hence, give the rate equation for the reaction and the units of the rate constant.

rate = $k\,[H^+][X][I_2]$

Units of k = $mol^{-2}dm^6\ s^{-1}$
…… [2]

(d) Suggest a safety precaution in this experiment.

As the concentration of the sulfuric acid is high, wear hand gloves to avoid direct contact.
…… [1]

[Data Processing] :*Kinetics Practical 4*

3. To investigate the effect of temperature on rate and E_a

This experiment investigates how the rate of the reaction between hydrochloric acid and sodium thiosulfate, $Na_2S_2O_3$, depends on the temperature and hence calculate a value for the activation energy of the following reaction.

$$S_2O_3^{2-}(aq) + 2H^+(aq) \rightarrow S(s) + SO_2(g) + H_2O(l)$$

Since the rate constant k is inversely proportional to time, t, the Arrhenius equation:

$$\lg k = \frac{-E_a}{RT} + C$$

may be written as $$\lg t = \frac{E_a}{2.303RT} + C$$

where E_a is activation energy, T is temperature in Kelvin and C is a constant.

Therefore, a plot of lg t against $\frac{1}{T}$ will be a straight line and the slope of the line is $\frac{E_a}{2.303R}$ where R = 8.31 $Jmol^{-1}K^{-1}$. Thus, the activation energy, E_a, for the reaction can be calcuated.

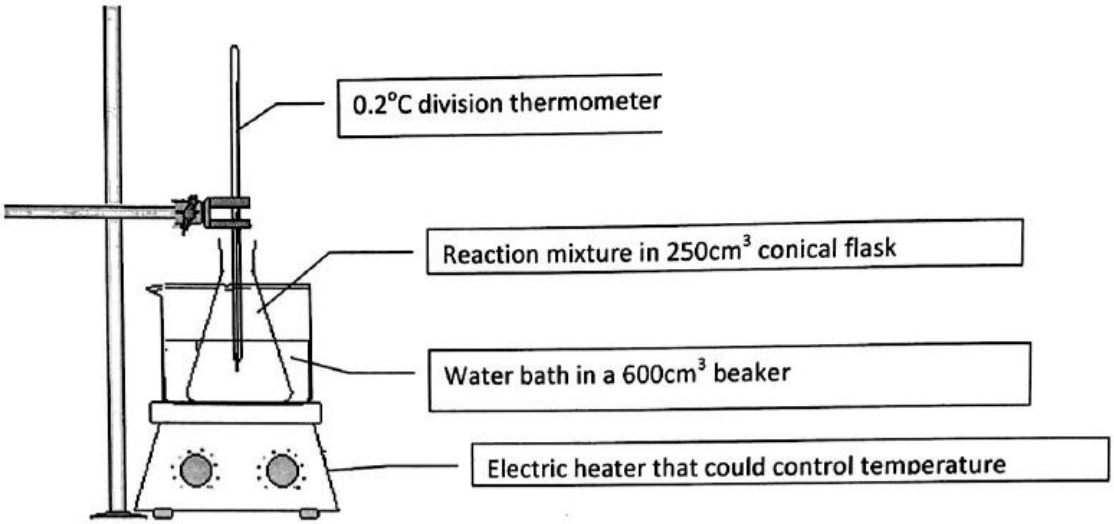
You are provided with the following:

0.100 mol dm^{-3} aqueous sodium thiosulfate
1.00 mol dm^{-3} hydrochloric acid

Procedure

1. Draw a large "X" mark using a marker on the surface of the hot plate.
2. Prepare a water bath using a 600 cm^3 beaker on the hotplate.
3. Add 100 cm^3 of 0.100 mol dm^{-3} $Na_2S_2O_3$ into a 250 cm^3 conical flask using a 100 cm^3 measuring cylinder.
4. Place the 250 cm^3 conical flask from step **3** into the water bath and carefully stir the solution until its temperature is between 70 ºC and 80 ºC.
5. Prepare 10 cm^3 of 1.00 mol dm^{-3} HC*l* using a 10 cm^3 measuring cylinder.
6. Pour the 10 cm^3 HC*l* solution rapidly into the conical flask containing $Na_2S_2O_3$ and start the stopwatch.
7. Stir the mixture using a glass rod and note the initial temperature, T_i of the reaction mixture using a 0.2 ºC division thermometer.
8. Stop the time when the "X" mark is completely obscured.
9. Note the final temperature T_f of the reaction mixture.
10. Note the time to the nearest second.
11. Wash the beaker and thermometer and dry them.
12. Repeat the experiment at temperatures ranging from 80 ºC to 40 ºC.

The experimental set-up is shown on the next page.



Results

Expt	T_i / °C	T_f / °C	T_{ave} / °C	T_k / K	t / s	$\frac{1}{T_K}$ / K^{-1}	lg t
1	42.0	48.0	45.0	318.0	180	3.14×10^{-3}	2.26
2	50.0	56.0	53.0	326.0	73	3.07×10^{-3}	1.86
3	64.0	68.0	66.0	339.0	16	2.95×10^{-3}	1.20
4	73.0	77.0	75.0	348.0	6	2.87×10^{-3}	0.778

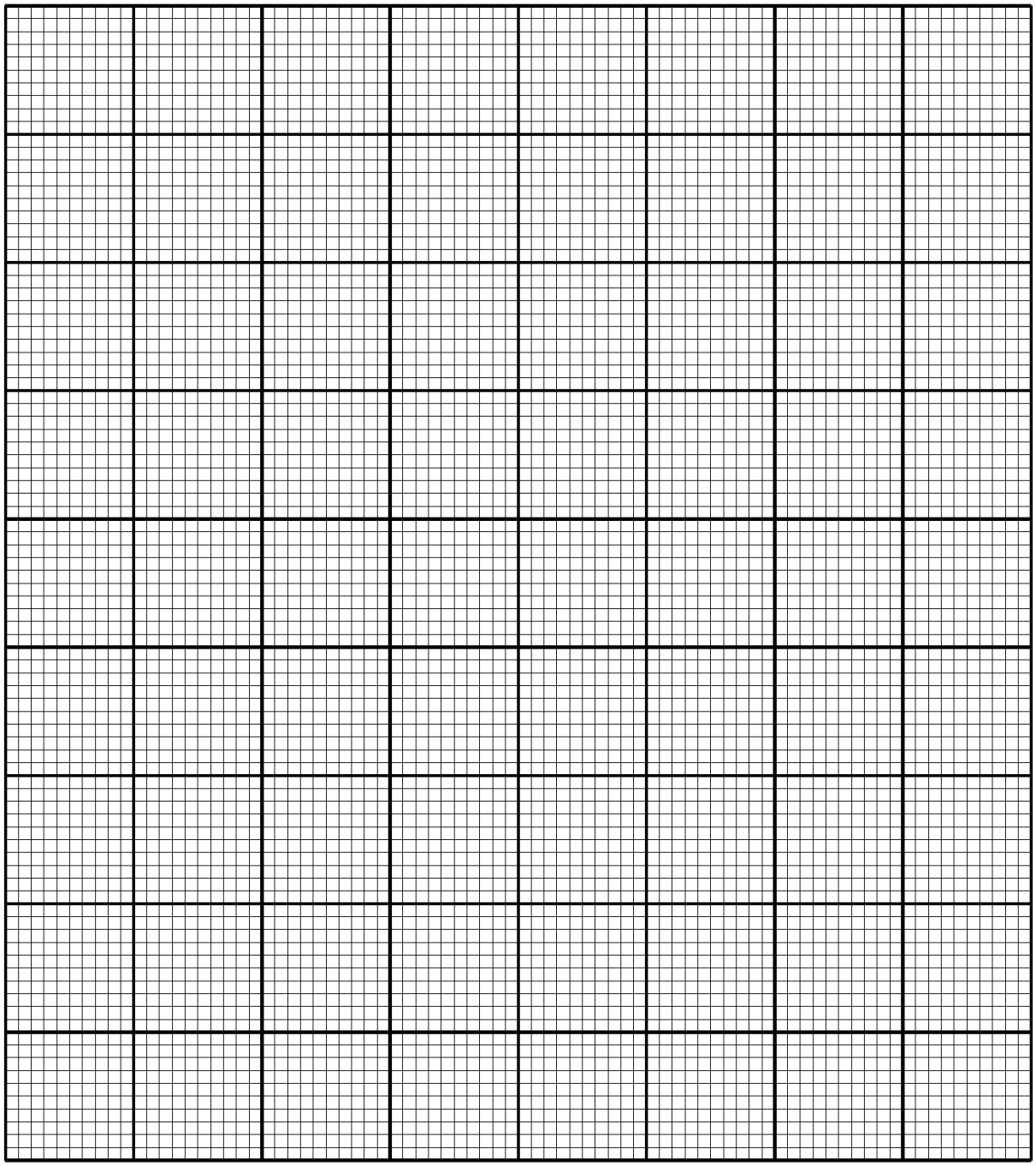
(a) Process the results in the table above to calculate both average temperature, T_{ave}, and temperature in Kelvin, T_K.

Include calculated values in the additional columns of the table that enable you to plot lg t against $\frac{1}{T_K}$. You may use some or all of these columns.

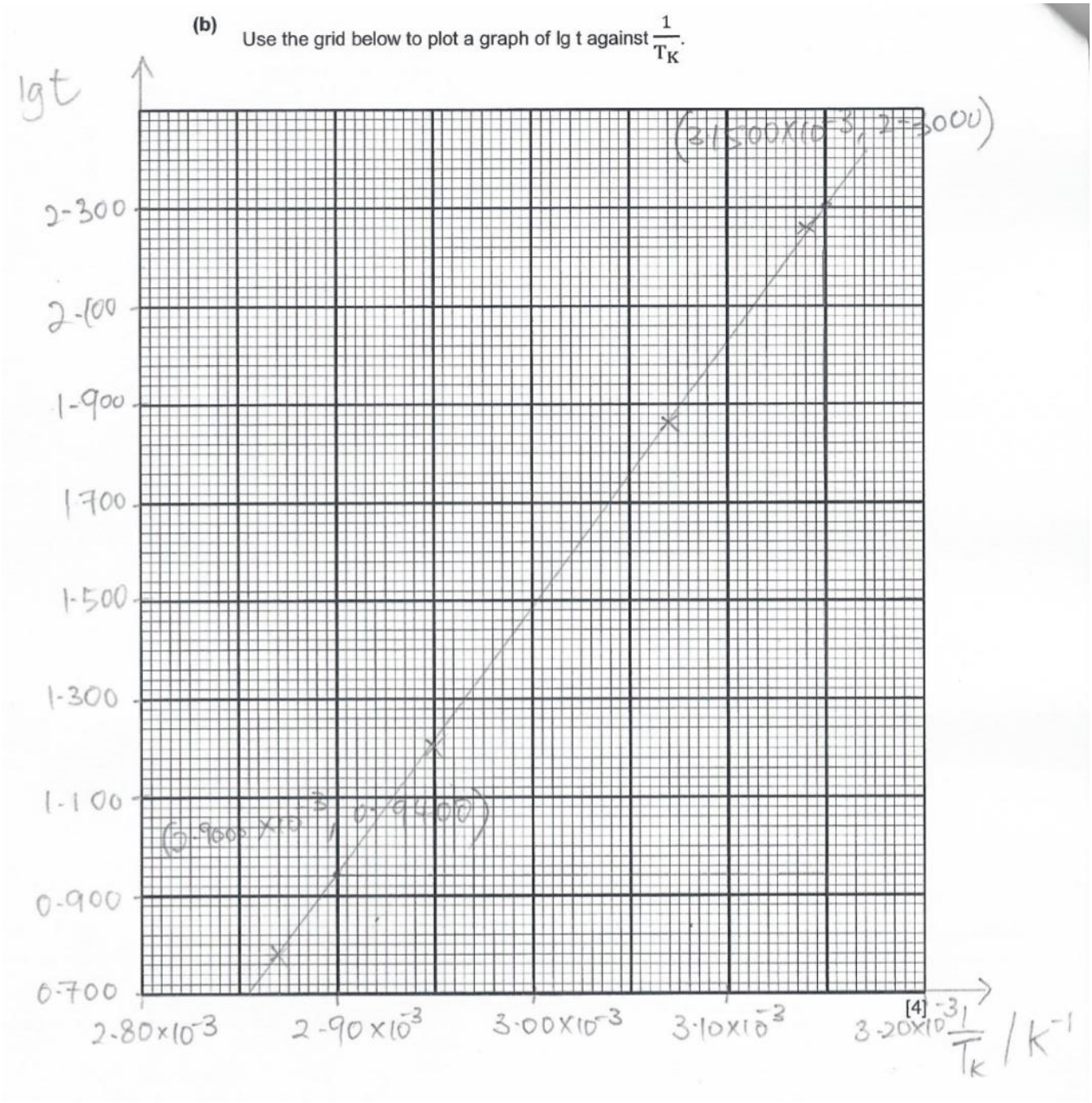
Label the columns you use clearly, including the units.

[3]

(b) Use the grid below to plot a graph of lg t against $\frac{1}{T_K}$.



(b) Use the grid below to plot a graph of lg t against $\frac{1}{T_K}$.



[4]

[4]

(c) Determine the gradient of the graph plotted in **(b)** and hence calculate a value for the activation energy of the reaction.

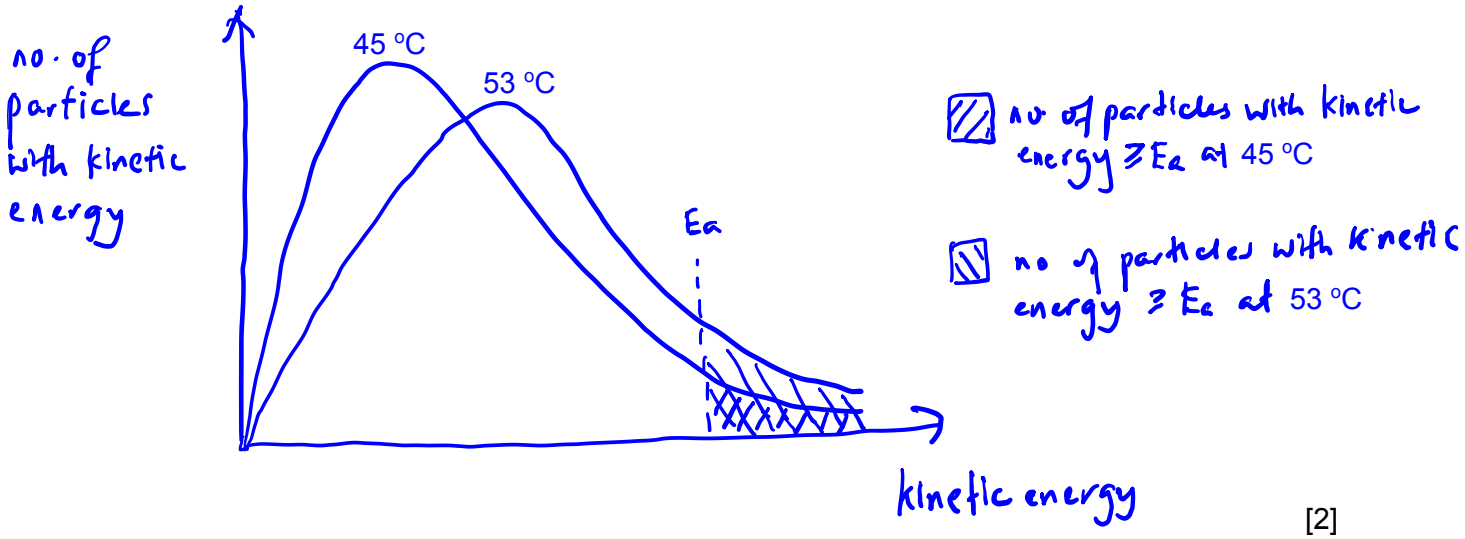
$$\text{Gradient} = \frac{y_1 - y_2}{x_1 - x_2} = \frac{2.3000 - 0.9400}{(3.1500 - 2.9000) \times 10^{-3}} = 5440$$

$$\frac{E_a}{2.303R} = \text{gradient of graph} = 5440$$

$E_a = 104 \text{ kJ mol}^{-1}$

activation energy = 104 kJ mol^{-1} ..... [2]

(d) **(i)** Draw the Boltzmann distribution curve for the reaction between hydrochloric acid and sodium thiosulfate at 45 °C and 53 °C.

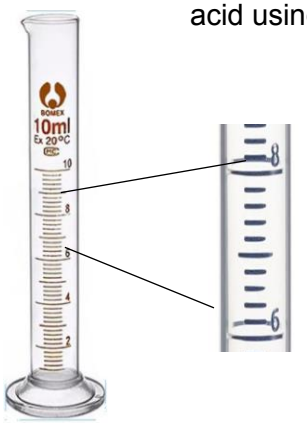


[2]

(ii) Hence, explain why the rate of reaction is faster at a higher temperature.

Average kinetic energy increases, and there are more particles with energy greater than or equal to the activation energy of the reaction at a higher temperature as compared to a lower temperature. Hence, there is a greater frequency of effective collision at a higher temperature and hence a faster rate of reaction.

(e) **(i)** Calculate the percentage uncertainty for the measurement of hydrochloric acid using a 10 cm^3 measuring cylinder.



$$\text{Percentage error} = \frac{\pm 0.1}{10} \times 100 = \pm 1.00\%$$

percentage error = ±1.00% ..... [1]

(ii) Suggest an improvement to reduce the percentage error obtained from **e(i)**.
Increase the volume of the hydrochloric acid used.

*Alternatively can use pipette or burette but can only start the stopwatch when solution is half-transferred (average of start time) as complete transfer of the solution to the reaction mixture takes a long time.

(f) State a hazard that must be considered when planning the experiment and describe precautions that should be taken to keep risks to a minimum.

Aqueous sodium thiosulfate solution and HC*l* may cause irritations/ skin corrosion from repeated or prolonged contact, hence wear safety gloves.

SO_2 gas is highly corrosive and can cause severe respiratory tract issues and eye and skin irritation. Carry out the experiment in a fume hood.

Data Processing

[Data Processing] : Inorganic *Qualitative Analysis Practical 1*

Instructions

For this experiment, you are to watch the video recordings on Google Classroom in order to record your observations.

1. At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs. Marks are **not** given for chemical equations. **No additional tests for ions present should be attempted.**

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) You are provided with a sample labelled **FA 5**. This sample **a mixture of 2 salts**, one of which contains Zn^{2+}. By carrying out the following tests you are able to suggest the identities of the ions present in **FA 5**.

Test	Observations
(i) Transfer the solid **FA 5** into a test tube and add a 3 cm depth of dilute nitric acid to make solution **FA 6**. Use this solution in tests **(ii)** to **(v)**. **Hint:** as 2 salts are present, 2 cations and 2 anions may be present. Both or either of the anions can produce gas with acid.	White solid dissolves in nitric acid to form a colourless solution. Gas evolved forms a white ppt with $Ca(OH)_2(aq)$. Gas does not decolourise purple $KMnO_4$. Note: Students should have written a colourless solution is formed as observed from video.
(ii) To a 1 cm depth of **FA 6** in a test-tube, add aqueous sodium hydroxide. **Hint:** Since **FA6** is a colourless solution, what are the other possible cations? See QA notes on pg 85-86. Think of how you can distinguish them.	white precipitate formed, soluble in excess NaOH(aq) to give a colourless solution.

(iii) To a 1 cm depth of **FA 6** in a test-tube, add aqueous ammonia.	white precipitate formed, insoluble in excess $NH_3(aq)$
Test	**Observations**
(iv) To a 1 cm depth of **FA 6** in a test-tube, add a 1 cm depth of aqueous silver nitrate,	No precipitate formed.
(v) To a 1 cm depth of **FA 6** in a test-tube, add a 1 cm depth of aqueous barium chloride or barium nitrate.	white precipitate formed, insoluble in excess $Ba(NO_3)_2$ (aq)

[5]

(vi) Suggest the identity of the cation (other than Zn^{2+}) and anions present in **FA 5**. Explain your choice.

Cation:	Al^{3+}. In test (a)(ii) and (iii), it gives a white precipitate of $Al(OH)_3$ with $NaOH/NH_3(aq)$ but only soluble in excess NaOH(aq) to give $[Al(OH_4)]^-$.
Anions:	SO_4^{2-}. In test (a)(i) and (v), it forms a white precipitate of $BaSO_4$ with acidified $Ba(NO_3)_2$ (aq). CO_3^{2-}. In test (a)(i), gas evolved forms a white precipitate with $Ca(OH)_2(aq)$ upon addition of nitric acid. CO_2 evolved.

[3]

(b) You are provided with solid **FA 7**, which is **a mixture of two salts**. Use separate portions of **FA 7** to perform the experiments below.

	Observation
(i) Heat a spatula measure of **FA 7** in a test-tube gently at first, then **Hint:** note the colour of the solid, may indicate presence of transition metal cation.	Condensation of water vapour occurs on cooler surface of test-tube. *at time 0.59min
heat more strongly and test the gas evolved with litmus paper. **Hint:** Decomposition of **polyatomic ion in solid** only applies to NH_4^+, CO_3^{2-} and NO_3^-.	Colourless, pungent gas evolved that turns damp red litmus blue

Dissolve a spatula measure of **FA 7** in a test-tube with a 5 cm depth of deionised water for use in tests **(ii)** and **(iii)**.	
(ii) To a 1 cm depth of **FA 7**(aq) in a test-tube, add aqueous silver nitrate,	White precipitate formed
followed by aqueous ammonia.	White precipitate soluble in excess NH_3 to give a colourless solution.
(iii) To a 1 cm depth of **FA 7**(aq) add barium chloride or barium nitrate,	White precipitate formed, insoluble in excess $Ba(NO_3)_2$ (aq).
followed by dilute nitric acid.	White precipitate dissolves in excess nitric acid to form a colourless solution. Effervescence observed. Colourless, odourless gas evolved formed a white precipitate with $Ca(OH)_2$.

[5]

(iv) Use your observations and the QA Table on pg 85-86 to identify **three** of the ions present. Give evidence for your choice of ions.

Ion	Evidence
NH_4^+	In test (b)(i), upon heating, a colourless pungent gas of NH_3 evolved, it turned damp red litmus blue.
Cl^-	In test (b)(ii), a white precipitate of AgC*l* is formed with $AgNO_3$ and the white precipitate dissolved in excess aq NH_3.
CO_3^{2-}	In test (b)(iii), a white precipitate of $BaCO_3$ is formed with $Ba(NO_3)_2$ and white precipitate dissolved in nitric acid to give off CO_2, which formed a white precipitate with $Ca(OH)_2$.

[3]

[Data Processing] : Inorganic *Qualitative Analysis Practical 2*

Instructions

For this experiment, you are to watch the video recording on Google Classroom in order to record your observations.

2. At each stage of any test you are to record details of the following.
- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs. Marks are **not** given for chemical equations. **No additional tests for ions present should be attempted.**

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) Some cations interfere with tests for anions and have to be removed from the solution before the tests for anions present can be performed. One way in which this can be carried out is to precipitate the cation in the form of its insoluble carbonate.

Test	Observation	
	FA 3	FA 5
To 5 cm depth of solution in a test tube, add all of the sodium carbonate, Na_2CO_3, from one of the tubes provided. Stir the mixture.	Blue / greenish-blue precipitate formed No effervescence is observed.	Green precipitate formed No effervescence is observed.

Retain the mixture from **FA 3** for use in **(b)**.

(b) Filter the mixture from **FA 3** from **(a)** into another test tube. Ignore any colour in the filtered solution. Add 5 cm depth of dilute nitric acid to the filtrate obtained. This removes any excess of carbonate ions.

Carry out the following tests on the acidified filtrate from **FA 3**.

Test	Observation
To 2 cm depth of the acidified filtrate from **FA 3** in a test-tube, add 1 cm depth of aqueous silver nitrate	White precipitate formed.
add an excess of aqueous ammonia.	White precipitate is soluble in excess aqueous ammonia to form a colourless solution.

[1]

(c) Carry out the following test on **FA 4**.

Test	Observation
To 1 cm depth of **FA 4** in a test-tube, add 1 cm depth of **FA 3**. **Hint:** if the ppt is formed in a coloured solution, allow time for the solid/ ppt to settle at the bottom so that colour of the solution can be observed more accurately.	A brown suspension formed. / Brown precipitate formed in a brown solution. White ppt in brown solution upon standing.
followed by adding a few drops of starch.	The brown suspension formed a dark blue / blue-black suspension.

[1]

(d) Carry out the following tests.

	Observations		
	FA 3	**FA 4**	**FA 5**
To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide. Next, add aluminium foil and warm cautiously. Test for any gas with a moist red litmus paper	Blue precipitate formed, which is insoluble in excess NaOH(aq) Moist red litmus paper remains red	White precipitate formed, which dissolves in excess NaOH to form a colourless solution Moist red litmus paper remains red	Green precipitate formed, which is insoluble in excess NaOH. Ppt exposed to air turns brown with time. Colourless and pungent gas evolved, which turned moist red litmus paper blue.
To 1 cm depth of solution in a test-tube, add aqueous ammonia.	Blue precipitate formed, which is soluble or dissolves in excess aq NH_3 to form a deep blue solution.	White precipitate formed, which is insoluble in excess NH_3.	Green precipitate. is formed, which is insoluble in excess NH_3. Ppt exposed to air turns brown with time.

[6]

(e) The results from tests in **(d)** should enable you to identify a sin cation in a solution.

solution	cation(s)
FA 3	Cu^{2+}
FA 4	Al^{3+}
FA 5	Fe^{2+}

[2]

(f) By considering the results of **all** your tests, enter one of the following responses in each of the boxes below.

- chloride
- iodide
- NO_3^-
- NO_2^-
- Insufficient test

FA 3	chloride
FA 4	iodide
FA 5	insufficient tests

Note: colourless iodide, I^- is typically used to test for ions with oxidising property, a positive test is indicated by the brown solution (I_2(aq))/ blue black coloration due to iodine-starch complex formed with starch indicator.

[2]

(g) Where you were not able to identify a single anion present in **(f)**, with reference to QA notes on pg 81-82, suggest a suitable reagent which would allow you to identify which anion is present in the solution from the list in **(f)**.

Do not carry out this test.

The reagent used is HCl /H_2SO_4.

(h) State the expected observation for the test in **(g)** to identify the presence of each of the anions.

If anion is NO_3^-, there will be no effervescence and no brown gas evolved.

Anion 1 ..... [1]

If anion is NO_2^-, effervescence observed and brown gas of NO_2 evolved.

Anion 2 ..... [1]

(i) When the test for **(a)** was repeated with Fe^{3+}, effervescence was observed. With the aid of two relevant equations with state symbols, explain why Fe^{3+} exhibits acidic properties and the gas produced upon addition of CO_3^{2-}(aq).

Fe^{3+} with high charge/size ratio undergoes hydrolyses in water to form H_3O^+:

$[Fe(H_2O)_6]^{3+}$ (aq) + H_2O (*l*) ⇌ $[Fe(H_2O)_5(OH)]^{2+}$ + H_3O^+ (aq)

which reacts with CO_3^{2-} to give CO_2, hence effervescence is observed and the gas evolved forms a white precipitate with limewater.

$2H_3O^+$ (aq) + CO_3^{2-}(aq) → CO_2(g) + $3H_2O$ (*l*)

Fe^{3+} with high charge/size ratio, has high polarising power. It weakens the O-H bond in water, causing the release of H^+ ion.