H2 Chemistry Preliminary Exam Practical Suggested Solution

1 Determination of the M_r of a hydrated ethandioate 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 $\mathbf{X}_2C_2O_4.H_2O$, where **X** is a Group 1 metal.

Solution **Q** contains 64.5 g dm^{-3} of $\mathbf{X}_2C_2O_4.H_2O$ in deionised water. You are not provided with **Q.**

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.

In this question, you will perform a titration. The data from this titration will be used to determine:

- the concentration of $C_2O_4^{2-}$ in **Q**,
- the M_r of $\mathbf{X}_2C_2O_4.H_2O$, and hence the identity of **X**.

(a) Titration of FA 1 against FA 2

In this titration, **FA 2** is run from the burette into the conical flask containing **FA 1** and **FA 3**. Initially, the colour of the **FA 2** will take some time to disappear.

After some **FA 2** has been added, sufficient $Mn^{2+}(aq)$ ions will be present to allow the reaction to occur faster.

The end-point is reached when a permanent pale pink colour is obtained.

(i)

1. Fill the burette with **FA 2**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 1** into the conical flask.
3. Using an appropriate measuring cylinder, transfer 50.0 cm^3 of **FA 3** to the same conical flask.
4. Heat this solution to about 65 °C.
5. Run **FA 2** from the burette into this flask until a **permanent** pale pink colour is obtained.
6. Record your titration results in the space provided on page 3. Make certain that your recorded results show the precision of your working.
7. Repeat points **1** to **6** as necessary until consistent results are obtained.
8. **Turn off your Bunsen burner**.

Results

Final burette reading/ cm^3	**25.10**	**25.10**
Initial burette reading/ cm^3	**0.00**	**0.00**
Volume of FA 2/$KMnO_4$/Titrant / cm^3	**25.10**	**25.10**
Values used (Tick consistent readings ±0.10 cm^3)	✓	✓

[5]

(ii) From your titrations, obtain a suitable volume of **FA 2** to be used in your calculations.

Show clearly how you obtained this volume.

Average volume of FA 2 = (25.10 + 25.10)/2 = 25.10 cm^3

Volume of **FA 2** =…25.10 cm^3….

[1]

(b) **(i)** The equation for the reaction between ethanedioate ions and manganate(VII) ions is shown below.

$5C_2O_4^{2-}(aq) + 2MnO_4^-(aq) + 16H^+(aq) \rightarrow 10CO_2(g) + 2Mn^{2+}(aq) + 8H_2O(l)$

Calculate the amount, in moles of ethanedioate ions, $C_2O_4^{2-}$ in 25.0 cm^3 of **FA 1**.

amount of MnO_4^- in FA 2 = (25.10/1000) X 0.02 = 0.000502 mol

amount of $C_2O_4^{2-}$ in 25 cm^3 of FA 1 = 0.000502/2 X 5 = 0.001255 mol

[1]

Amount of $C_2O_4^{2-}$ in 25 cm^3 of **FA 1** =…0.001255 mol or 0.00126 mol…

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

amount of $C_2O_4^{2-}$ in 250 cm^3 of FA 1 = 0.001255 X 10 = 0.01255 mol

$[C_2O_4^{2-}]_{in\ Q}$ = 0.01255 / (35.70/1000) = 0.3516 mol dm^{-3}

[2]

Concentration of $C_2O_4^{2-}$ in **Q** =…0.3516 or 0.352 mol dm^{-3}

(iii) Use your answer to **(b)(ii)** to calculate the M_r of the ethanedioate salt.

M_r of $\mathbf{X}_2C_2O_4.H_2O$ = 64.5 / 0.3516 = 183.4

M_r of the ethanedioate salt = 183.4 ……

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]

Ar of X = [183.4 – 2(12.0) – 5(16.0) – 2(1.0)]/2 = 38.7

X = Potassium

[3]

X is K……

1 (c) A student performed the experiment in **(a)(i)** using a sample of another ethanedioate salt. The student obtained a mean titre value of 22.20 cm^3. The teacher calculated that the volume of **FA 2** required should have been 22.40 cm^3. The teacher told the student that the total percentage error from the apparatus in the experiment was 0.4 %.

Calculate the error in the student's result, based on these data. State **and** explain whether or not the student's result is accurate.

Student's experimental error = $\frac{22.40 - 22.20}{22.40} \times 100\% = 0.8928 = 0.893\%$

Since student's experimental error (0.893%) is more than the apparatus error(0.4%), therefore the student's result is inaccurate.

[2]

[Total: 14]

2 Evaluation of the reliability of a gas collection method in determining a value for the M_r of the ethanedioate salt.

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.

FA 4 is a solution containing manganese(II) ions, Mn^{2+}.

You will need access to the **FA 1**, **FA 2** and **FA 3** solutions you used earlier.

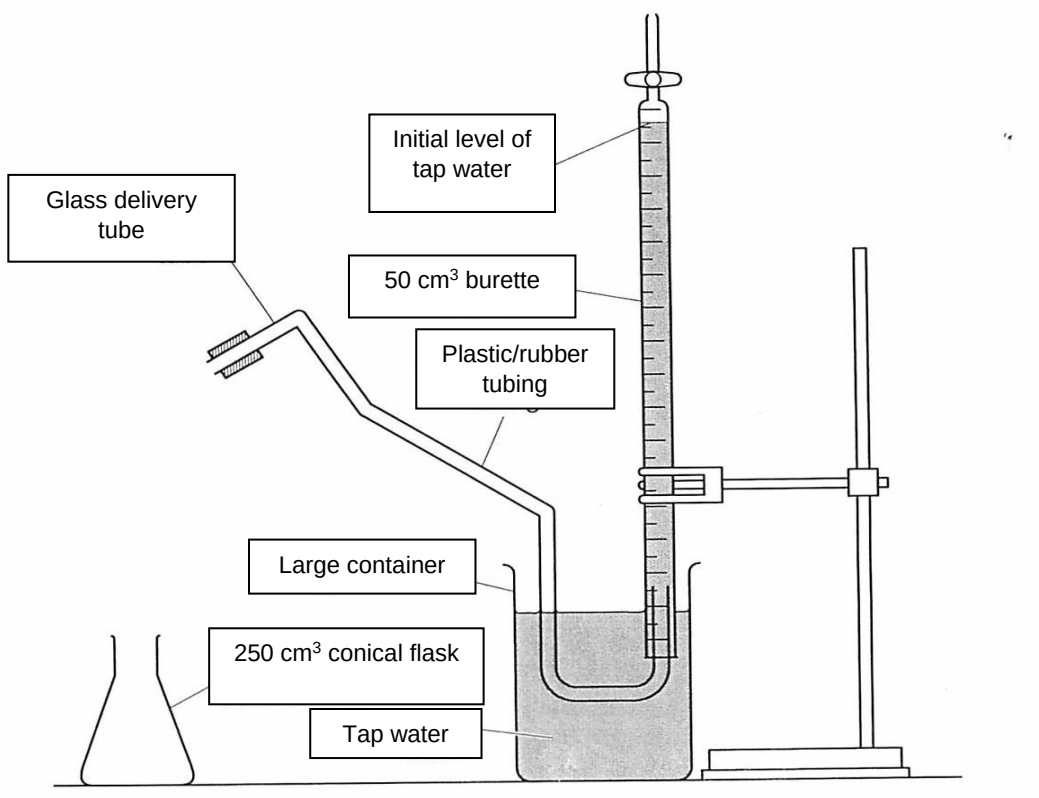


Fig. 2.1

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

In an appropriate format in the space provided on page 7, prepare a table in which you may record each burette reading and the time it was taken.

In addition, your table 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. 2.1. You should insert the plastic/rubber tubing to a sufficient depth 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. Use 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.
8. 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.
9. Check that the plastic/rubber tubing is securely positioned in the burette.
10. Hold the flask by its neck and gently swirl it continuously.
11. 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.
12. Continue to gently swirl the flask. Read and record the water level in the burette every half minute, until the reaction is complete.

(a) (i) Experimental Results

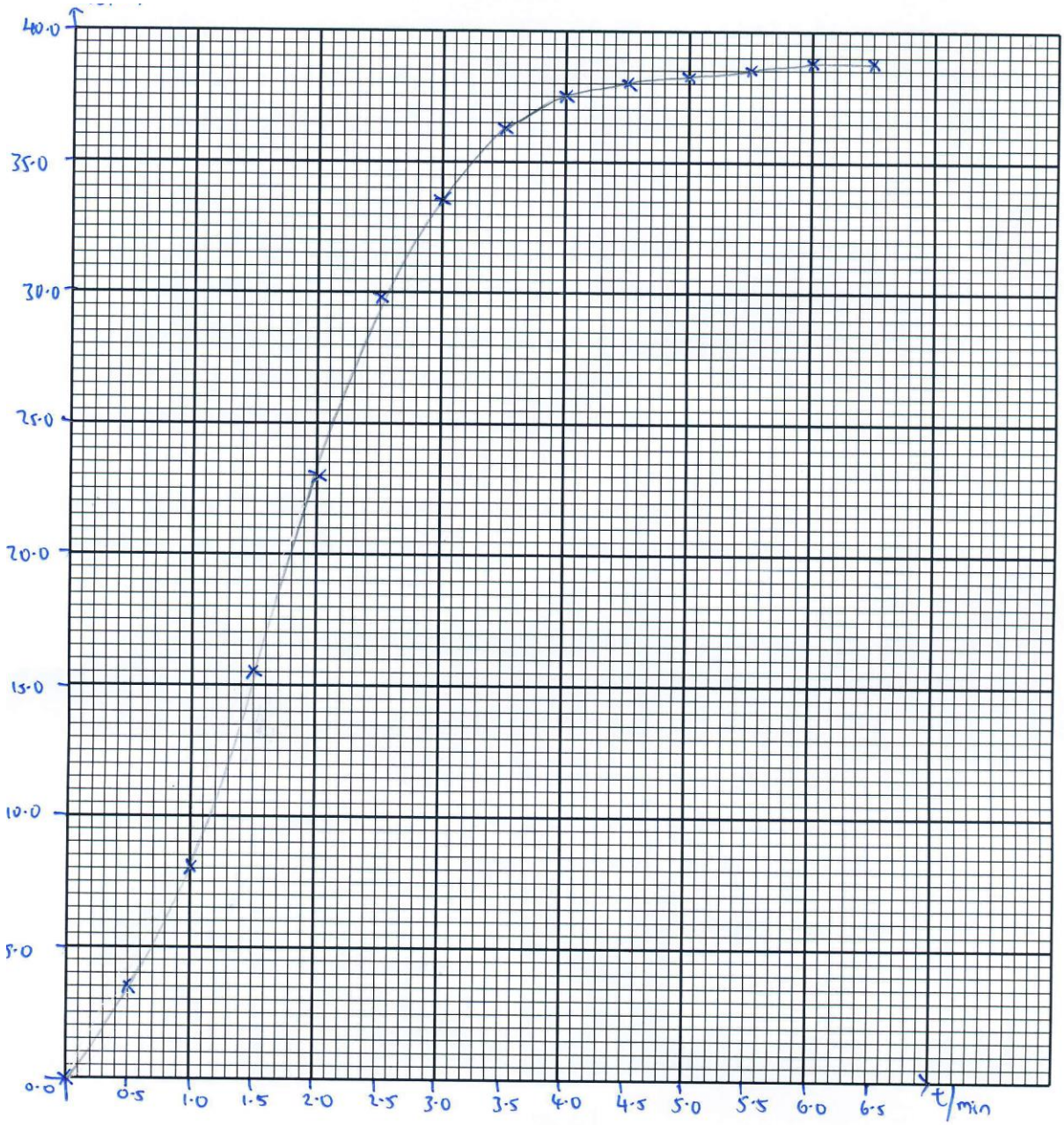
Time / t / min	Burette reading / cm^3	(Volume / vol) of CO_2 / cm^3
0.0	50.0	0.0
0.5	46.5	3.5
1.0	42.0	8.0
1.5	34.5	15.5
2.0	27.0	23.0
2.5	20.2	29.8
3.0	16.5	33.5
3.5	13.8	36.2
4.0	12.5	37.5
4.5	12.0	38.0
5.0	11.6	38.4

5.5	11.5	38.5
6.0	11.4	38.6
6.5	11.4	38.6
7.0	11.4	38.6

[3]

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

Vol of CO_2 /cm^3 Draw the most appropriate line, taking into account all of your points.



[4]

2 (a) (iii) The equation for the reaction between ethanedioate ions and manganate(VII) ions is shown below.

$5C_2O_4^{2-}(aq) + 2MnO_4^-(aq) + 16H^+(aq) \rightarrow 10CO_2(g) + 2Mn^{2+}(aq) + 8H_2O(l)$

Use this equation, and appropriate data from your graph, to calculate a value for the amount, in moles of ethanedioate ions, $C_2O_4^{2-}$, present in 20.0 cm^3 of **FA 1**.

[molar volume of gas = 24.0 $dm^3\,mol^{-1}$ at r.t.p.]

From graph, max volume of CO_2 = 38.55 cm^3

amount of CO_2 produced = 38.55 / 24000 = 0.001606 mol

amount of $C_2O_4^{2-}$ in 20.0 cm^3 of FA1 = 0.001606 / 2 = 0.000803 mol

Amount of ethanedioate ions, $C_2O_4^{2-}$, in 20.0 cm^3 of **FA 1** = 0.000803 mol **[1]**

(b) (i) Using your answer to question **1(b)(i)**, calculate the amount of ethanedioate ions in 20.0 cm^3 of **FA 1**. Hence, 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 $C_2O_4^{2-}$ in 20 cm^3 of FA 1 = 0.001255 / 25 X 20 = 0.0001004 mol

amount of CO_2 produced = 0.0001004 X 2 = 0.002008 mol

maximum volume of CO_2 = 0.002008 X 24000 = 48.19 cm^3

maximum volume of CO_2 produced from 20.0 cm^3 of **FA 1** = 48.2 cm^3 **[4]**

(ii) 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)(i).**

Some of the CO_2 could have stayed dissolved in the water bath/Some CO_2 could have escaped when the conical flask was stoppered. **[1]**

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

Uses a non-aqueous gas collecting system such as a frictionless gas syringe. **[1]**

Use a dropping funnel to add in **FA2** to start the reaction to minimise the escaping of gas.

2 **(b)** **(iv)** In Question **1** you calculated a value for the M_r of the ethanedioate salt. The total volume of CO_2 collected in **2(a)(i)** could also be used to calculate a value for the M_r of the ethanedioate salt.

Suggest which of these two M_r values would be higher. Explain your answer.

The M_r calculated from the collection of gas method will be higher as the volume measured will be lower than expected, causing the number of moles of salt to be lower and hence the M_r to be higher. **[1]**

(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.

(i) 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 in **Fig. 2.2**.

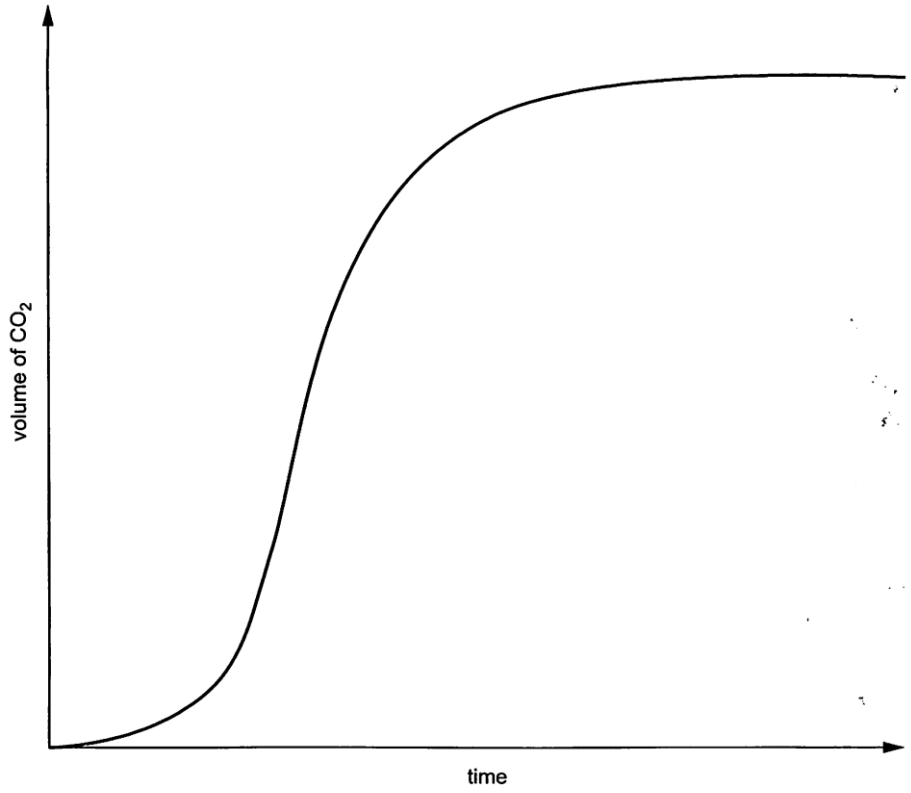


Fig. 2.2

2 (c) (i) Consider the **shape** of the graph in **Fig 2.2** and your graph in **2(a)(ii)**. Describe one major difference between the shapes. Suggest an explanation for your answer.

Difference The graph in 2(a)(ii) is steeper at the start of the experiment.

Explanation Mn^{2+} was added right from the start and hence the reaction was catalysed right from the beginning unlike the student's experiment where there were little Mn^{2+} at the beginning to catalyse the reaction, causing the student's initial gradient to be gentler. **[2]**

(ii) For the titration in **1(a)(i)** between ethanedioate ions, $C_2O_4^{2-}$, and manganate(VII) ions, MnO_4^-, the solution needs to be at about 65 °C at the start. As cold **FA 2** is added, the temperature of the mixture decreases. However, this decrease in temperature does not cause the rate of reaction between $C_2O_4^{2-}$ ions and MnO_4^- ions added from the burette to decrease. Suggest an explanation for this.

The stable rate is due to Mn^{2+} ions being produced. The presence of the Mn^{2+} catalyst cancels out the effect of the drop in temperature.

[1]

2 (d) Planning

The oxidation of iodide ions, I^-, by peroxodisulfate ions, $S_2O_8^{2-}$, is known to be catalysed by Fe^{2+} or Fe^{3+} ions.

A similar reaction, shown below, in which ethanedioate ions, $C_2O_4^{2-}$, are oxidised by peroxodisulfate ions, $S_2O_8^{2-}$, may be catalysed by Cu^{2+} ions and by Ag^+ ions.

$$S_2O_8^{2-} + C_2O_4^{2-} \rightarrow 2\ SO_4^{2-} + 2\ CO_2$$

(i) Suggest an explanation why both of these reactions are slow when performed in the absence of a catalyst.

The reactants are both negatively charged and experience repulsion and thus resulted in a high activation energy barrier between them.

[1]

(ii) Plan an investigation to test how well, **if at all**, Cu^{2+} ions and by Ag^+ ions catalyse the reaction between $C_2O_4^{2-}$ ions and $S_2O_8^{2-}$ ions.

In your plan, you should include details of:

- the reactants and conditions that you would use,
- the apparatus you would use and the procedure you would follow,
- the measurements you would take,
- an outline of how you would use your results to compare the effectiveness of each ion as a potential catalyst.

Suggested Planning Procedure

Note: It was assumed that the temperature and pressure conditions for the conduct of the 3 experiments is at rtp conditions

Experiment 1 (uncatalysed reaction)

1. Using a 50 cm^3 measuring cylinder, measure out 15 cm^3 of **$X_2C_2O_4$ of concentration 0.05 $moldm^{-3}$** into a 250 cm^3 conical flask. Add 5 cm^3 of deionised water using a dropping pipette (or 10 cm^3 measuring cylinder).
2. Use a separate 50 cm^3 measuring cylinder, measure out 20 cm^3 of a solution of $Na_2S_2O_8$ of concentration 0.05 $moldm^{-3}$.
3. Set up the apparatus as shown in Fig. 2.1
4. Check that the plastic/rubber tubing is securely positioned in the burette.
5. Transfer the 20 cm^3 of a solution of $Na_2S_2O_8$ into the conical flask and insert the bung into the conical flask. (Using dropping funnel to transfer 20 cm^3 of solution will be better to minimise gas escape.)
6. 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.
7. Hold the flask by its neck and gently swirl it continuously.
8. 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.
9. Continue to gently swirl the flask. Read and record the water level in the burette every half minute, until the reaction is complete. i.e. at least 3 burette readings are the same.
   (Estimated volume of CO_2 is 36 cm^3)

Pre-Calculation:

No. of moles of $C_2O_4^{2-}$ (limiting agent) = 0.05x15/1000 = 0.00075 mol

Volume of CO_2 produced

= 0.00075 X 2 X 24000 = 36 cm^3 assuming r.t.p.

Catalysed Experiment 2 and 3

10. Repeat steps 1 to 9 but now with the introduction of the catalysts Ag^+ (Experiment 2) and Cu^{2+} (Experiment 3), once at each time. Do take note that 5 cm^3 of each catalyst (of equal concentration 0.100 mol dm^{-3} is to be added in **step 1**.
11. Compare the time taken for the maximum amount of CO_2 to be produced for Experiment 1 (uncatalysed reaction) with Experiment 2 (Ag^+ as catalyst) and Experiment 3 (Cu^{2+} catalyst). The better catalyst

will be the one that produces the maximum amount of CO_2 in the shortest period of time.

Or Plot the graph of the volume of CO_2 against the time taken. The more effective catalyst is determined by the catalyst that produce a steeper gradient.

Alternatively,

9. Record the time required to produce 20 cm^3 (a stated volume, based on pre-calculation) of CO_2. The more effective catalyst is determined by the catalyst that produce the volume in a shorter time.

[7]

[Total: 26]

3 You are provided with the solid **K12** which contains one cation given in the Qualitative Analysis Notes.

You are to perform the tests below to identify the cation present in **K12** and suggest the nature of **K12.** Record your observations in the spaces provided. Your answers should include

- Details of colour changes and precipitates formed,
- The names of any gases evolved and details of the test used to identify each one.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional or confirmatory tests for ions present should be attempted.

	Tests	Observations and Deductions
1	Heat **K12** alone.	K **12** turns to a colourless liquid. Gas evolved turns moist blue litmus red Gas evolved turns acidified $KMnO_4$ paper colourless SO_2 (g) Gas relights glowing splinter O_2(g)
2	Warm **K12** with dilute hydrochloric acid. Keep the solution.	Gas evolved turns moist blue litmus red then bleaches Cl_2 (g) Oxidising agent present

3	To the solution from test **2**, add dilute nitric acid and barium nitrate(V) solution	White ppt forms, insoluble in acid. SO_4^{2-} $BaSO_4$ ppt
4	Warm **K12** with a freshly made solution of iron(II) sulfate solution.	Pale green solution turns brown/ red brown K **12** is an oxidising agent
5	Dissolve **K12** in dilute nitric acid. Add manganese(II) sulfate solution and two drops of silver nitrate(V) solution to act as a catalyst. Boil the mixture.	Pale pink colour turns brown then to black/purple solution (ppt) Mn^{2+} oxidise to MnO_2 then to MnO_4^- K **12** is an oxidising agent
6	Warm **K12** with sodium hydroxide solution.	Colourless solution obtained. Pungent smell gas which turns moist red litmus blue NH_3 gas NH_4^+ present

Nature of **K12:** **Oxidising agent**

Cation it contains: NH_4^+

[9]

[Total: 9]

4 Planning

You are provided with 3 unlabelled bottles and each bottle contains one of the following organic compounds,

- Butanamide
- Lactic acid, $CH_3CH(OH)CO_2H$
- Pyruvic acid, CH_3COCO_2H

(a) Outline a sequence of simple chemical tests to identify each of the above organic substances. You may use any other common laboratory chemical reagents and apparatus, if necessary. You are **not** allowed to identify the substances by elimination. You are reminded that most of the compounds listed are *flammable* liquids. **[3]**

Test	Expected Observations & Deductions
Using a dropper, add 1 cm^3 of compound into 3 separate test-tubes. To 1 cm^3 of compound in a test-tube, add 2 cm^3 of aqueous NaOH. Boil/ Heat in a **water bath** for 5-10 min. Test any gas evolved with moist red litmus paper	**Moist red litmus paper turns blue (NH_3) - butanamide**
To the 2 samples that did not produce a positive observation for test 1, using a dropper to 1 cm^3 of compound in a test-tube, add 1 cm^3 of 2,4 DNPH dropwise until excess. Warm the mixture.	**Orange ppt forms. – pyruvic acid**
To 1 cm^3 of the compound in a test-tube, add 1 cm^3 of dilute sulfuric acid, follow by a few drops of $K_2Cr_2O_7(aq)$. Heat the mixture in a hot water-bath for 3 -5 min.	**Orange solution turns green-lactic acid**
Alternatives: pyruvic acid can be tested with 2,4 DNPH, warm ➔ lactic acid with aqueous Na_2CO_3 ➔ butanamide with NaOH(aq), heat.	

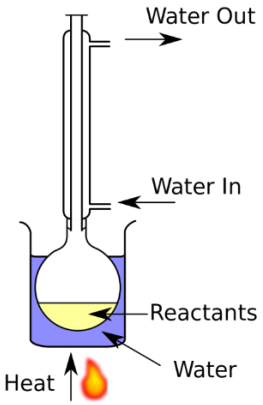
(b) Suggest a safety measure that you would consider in carrying out your plan.

Using a hot water bath for heating instead of using a direct naked flame from the Bunsen burner as most organic compounds are highly flammable.

[1]

(c) State the required reagents and conditions. Draw a set-up of the apparatus for the synthesis of pyruvic acid from lactic acid.

$KMnO_4(aq)/H_2SO_4(aq)$, heat under reflux OR $K_2Cr_2O_7(aq)/ H_2SO_4(aq)$, heat under reflux



[2]

[Total: 6]

The End