CJC CJC CJC CJC CJC CJC CJC CJC	Catholic Junior College JC2 Preliminary Examinations Higher 2
CANDIDATE NAME	
CLASS	2T

CHEMISTRY

Paper 4 Practical

23 August 2023 2 hours 30 minutes

9729/04

Candidates answer on the Question Paper. Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name and class in the boxes above.

Give details of the practical shift and laboratory, where appropriate, in the boxes provided. Write in dark blue or black pen.

You may use an HB pencil for any diagrams or graphs.

Do not use staples, paper clips, glue or correction fluid.

Answer **all** questions in the spaces provided on the Question Paper.

The use of an approved scientific calculator is expected, where appropriate. You may lose marks if you do not show your working or if you do not use appropriate units.

Qualitative Analysis Notes are printed on pages 21 and 22.

At the end of the examination, fasten all your work securely together. The number of marks is given in brackets [] at the end of each question or part question.

MARK SCHEME

Shift
Laboratory

For Examiner's Use		
1	/ 11	
2	/ 18	
3	/ 16	
4	/ 10	
Total	/ 55	

This document consists of **21** printed pages and **1** blank page.

Answer all the questions in the spaces provided.

1 Qualitative analysis of an inorganic solid mixture FA 8 is $MnCO_3 + Al(NO_3)_3$

FA 8 is a solid mixture that contains two cations and two anions listed in the Qualitative Analysis Notes on pages 21 and 22.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured.

- 1. Transfer the **FA 8** provided into a boiling tube and add in 3 cm depth of deionised water.
- 2. Using a glass rod, stir the mixture well for one minute until no more of the solid mixture can dissolve.
- 3. Filter the mixture into another clean boiling tube. While waiting, proceed to Question 2 first.
- 4. When the filtration is completed, place the filter funnel containing the residue on a conical flask and **wash the residue** with a little deionised water.

Keep both filtrate and residue for tests (a) and (b) below.

Test and identify any gases evolved.

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

If there is no observable change, write **no observable change**.

Record your observations in Tables 1.1 and 1.2 below. **No additional tests for ions present should be attempted.**

(a) Tests on the filtrate.

	test	observations
(i)	Test the filtrate using Universal indicator paper.	Universal indicator paper <u>turns yellow.</u>
	Hence conclude nH of the filtrate	pH 3 to 5
		Make sure the colour of the UI paper corresponds to the pH given. pH 3 – orange pH 4 – yellow pH 5 – yellowish-green

(ii)	To 1 cm depth of the filtrate in a test- tube, add aqueous sodium hydroxide dropwise, with shaking, until no further change is observed.	 <u>Off-white / light brown</u> ppt formed; <u>insoluble in</u> <u>excess</u> NaOH
		<u>Solubility</u> of ppt in <u>excess</u> NaOH must be included since question stated 'until no further change is observed'.

(b) Tests on the residue.

Table 1.2

	test	observations	
(i)	Place the funnel containing the residue (on the filter paper) into a clean boiling tube.	•	Effervescence observed; gas gives white ppt with limewater. CO2(g) liberated.
	Pour about 5 cm ³ of dilute nitric acid onto the residue. Collect 1 cm depth of the solution in the boiling tube. Remove the funnel and return it to the conical flask.	•	<text></text>

	included since question stated 'until no further change is observed'.		
(ii)	To this solution in the boiling tube, add aqueous sodium hydroxide dropwise, with shaking, until no further change is observed.	• <u>Off-white / beige / pale</u> <u>or light brown/cream</u> <u>ppt insoluble in excess</u> <u>NaOH(aq)</u> .	
	Keep this resultant mixture for test (iii) below.	 Ppt rapidly <u>turns brown</u> on contact with air / darkens on standing. 	
(iii)	To 1 cm depth of the mixture from (ii), carefully add 1 cm depth of aqueous hydrogen peroxide.	 ppt turns <u>brown / darker</u> <u>brown / brown-black</u>. Brisk <u>effervescence: gas</u> <u>re-ignite a glowing splint;</u> <u>O₂ gas liberated.</u> 	
		Test of gas and identity of gas must be included when effervescence is observed.	

Λ

(c) (i) One of the cations in **FA 8** is responsible for the nature of the filtrate in test **1(a)(i)**. It is formed from a Period 3 element.

This cation present in **FA 8** is: .Al³⁺.....

As the solution is acidic shown by UI paper (pH 3-5), it must be Al^{3+} . Mg²⁺ is not accepted because the solution is almost neutral (pH 6.5)

Solubility of ppt in excess NaOH must be

(ii) Write an equation to account for the nature of the filtrate in test 1(a)(i).

 $[Al(H_2O)_6]^{3+} + H_2O \rightleftharpoons [Al(H_2O)_5(OH)]^{2+} + H_3O^+$ [1]
Double arrow must be used to show that partial hydrolysis is occurring.
The second cation present in EA 8 is Mn^{2+}

(d) (i) The second cation present in **FA 8** is^{Mn²⁺}

evidence In test 1b(ii), addition of NaOH (aq) results in formation of a off-white

ppt that was insoluble in excess NaOH(aq). The ppt rapidly turned brown upon

contact with air, indicating presence of Mn²⁺. [1]

Reference to the test and observations must be included in the evidence to support the cation identified.

(ii) One anion present in FA 8 is CO_3^{2-}

evidence In test 1b(i), addition of nitric acid results in brisk effervescence of

<u>CO₂ gas which gave a white ppt with Ca(OH)₂, indicating presence of CO_3^{2-} .</u>

Reference to the test and observations must be included in the evidence to support the cation identified.	
	[1]

(iii) Suggest what type of reaction takes place when hydrogen peroxide is added in test

1(b)(iii).

Redox	[1]
	[Total: 11]

The reaction that is occurring here $Mn(OH)_2 + H_2O_2 \rightarrow MnO_2 + 2H_2O$ Off-white ppt brown-black ppt. $Mn(OH)_2$ reacted with H_2O_2 & oxidised to form brown-black MnO_2 solid. Redox is occurring because Mn^{2+} is oxidized to MnO_2 while H_2O_2 is being reduced to H_2O .

Explanation for O₂ liberated: The MnO₂ product catalysed the disproportionation of H₂O₂, H₂O₂ \Rightarrow 2H₂O + $\frac{1}{2}$ O₂ resulting in formation of H₂O(I) and O₂(g) which explain for the effervescence observed.

2 Determination of the molar enthalpy changes of two reaction by indirect methods.

Sodium carbonate, Na₂CO₃ reacts with water and carbon dioxide to form sodium hydrogencarbonate, NaHCO₃, according to equation 1.

equation 1 Na₂CO₃(s) + H₂O(I) + CO₂(g)
$$\rightarrow$$
 2NaHCO₃(s) ΔH_1

The enthalpy change of this reaction cannot be determined directly.

Both Na₂CO₃(s) and NaHCO₃(s) react with dilute sulfuric acid.

equation 2Na2CO3(s) + H2SO4(aq) \rightarrow Na2SO4(aq) + H2O(l) + CO2(g) ΔH_2 equation 3NaHCO3(s) + ½ H2SO4(aq) \rightarrow ½ Na2SO4(aq) + H2O(l) + CO2(g) ΔH_3

In this experiment, you will determine the values for ΔH_2 and ΔH_3 , and then use your results to calculate a value for ΔH_1 .

You are provided with:

FA 1 is sodium carbonate, Na₂CO₃.

FA 2 is sodium hydrogen carbonate, NaHCO₃.

FA 3 is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄ (**FA 3** will also be used in **Q3**).

(a) Method

Experiment 1: $Na_2CO_3(s) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + H_2O(l) + CO_2(g)$

- 1. Use a measuring cylinder to transfer 25 cm³ of the acid, **FA 3**, into a polystyrene cup supported in a 250 cm³ beaker. The acid is in excess.
- 2. Weigh the capped bottle containing **FA 1** and record the balance reading.
- 3. Place the thermometer in the acid and record its initial temperature.
- 4. Carefully tip all the FA 1, in small portions, into the acid and stir to dissolve.
- 5. Read and record the highest temperature reached.
- Reweigh the emptied bottle and its cap and record the balance reading and the mass of FA 1 used.

Experiment 2: NaHCO₃(s) + $\frac{1}{2}$ H₂SO₄(aq) $\rightarrow \frac{1}{2}$ Na₂SO₄(aq) + H₂O(l) + CO₂(g)

- 1. Replace the wet polystyrene cup with a clean, dry polystyrene cup.
- 2. Repeat Steps 1 to 4 in Experiment 1 but use FA 2 in place of FA 1.
- 3. Read and record the lowest temperature reached.
- 4. Reweigh the emptied bottle and its cap and record the balance reading and the mass of **FA 2** used.

Results

Record **all** weighings and temperature readings to an appropriate level of precision in the table below.

Table 2.1		
	Experiment 1	Experiment 2
mass of capped bottle + solid / g	7.599	10.625
mass of capped bottle + residual solid / g	5.607	8.129
mass of solid added / g	1.992	2.496
initial temperature, <i>T</i> _i , of FA 3 / °C	28.0	28.0
final temperature, T _f / °C	34.0	20.3
temperature change, $\Delta T / °C$	6.0	-7.7

all mass recorded consistently to 2 or 3 d.p. + all temperature readings recorded to 1 d.p. + ΔT and mass of solid used correctly calculated.

Take note the number of dp for all weighing must be the same throughout.

(b) For the purpose of calculations, you should assume that the specific heat capacity of the solution is 4.18 J $g^{-1}K^{-1}$, and that the density of the solution is 1.0 g cm⁻³.

Use relevant results from Table 2.1 to calculate the heat energy produced when (i) **FA 1** was added to the acid in **Experiment 1**. Hence, calculate the enthalpy change, in kJ mol⁻¹, when 1 mol of **FA 1**, Na₂CO₃, reacts with the acid. Include the sign of ΔH_2 in your answer.

[A_r: C, 12.0; O, 16.0; Na, 23.0].

heat

evolved = mc
$$\Delta T$$

= 25 × 4.18 × 6.0 25 × 4.18 × ΔT_{expt1}
= 627 J

The m here refers to the mass of the solution, not the solid. **AT refers to CHANGE of temperature. There is** no need to convert from °C to K.

627 J heat energy produced =.....

-33.4

*M*_r of Na₂CO₃ = 2(23.0) + 12.0 + 3(16.0) = 106.0
mol of Na₂CO₃ =
$$\frac{1.992}{106.0}$$
 = 0.0188 mol
∴ $\Delta H_2 = -\frac{627}{0.0188}$ Jmol⁻¹ ecf, & with correct sign

$$= - 33.4 \text{ kJ mol}^{-1}$$

enthalpy change,
$$\Delta H_2 = \dots -33.4$$
 kJ mol⁻¹

[3]

[5]

As temperature increase in experiment, this is **EXOTHERMIC** reaction. NEGATIVE SIGN must be included.

7

(ii) Use relevant results from **Table 2.1** to calculate the heat energy absorbed when **FA 2** was added to the acid in **Experiment 2**. Hence, calculate the enthalpy change, in kJ mol⁻¹, when 1 mol of **FA 2**, NaHCO₃, reacts with the acid. Include the sign of ΔH_3 in your answer.

[*A*_r: H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

heat absorbed = mc ΔT = 25 × 4.18 × 7.7 25 × 4.18 × ΔT_{expt2} = 804.6 J

As temperature decrease in experiment, this is ENDOTHERMIC reaction. POSITIVE SIGN must be included.

enthalpy change, $\Delta H_3 = \dots + 27.1$ kJ mol⁻¹ [3]

(iii) Using your answers to (b)(i) and (b)(ii) and the equations for Experiment 1 and Experiment 2, construct an energy cycle to determine a value for the enthalpy change for the reaction:

enthalpy change, $\Delta H_1 = \dots - 87.6$ kJ mol⁻¹ [2]

Correct energy cycle includes

- Balanced equations
- State symbols
- Correct identity and coefficient of
 \U00e4H

- (c) An additional experiment must be carried out using FA 2, solid sodium hydrogencarbonate, NaHCO₃, and deionised water to determine the enthalpy change of solution of NaHCO₃, ΔH_4 .
 - (i) Carry out the additional experiment using between 2.0 to 2.5 g of **FA 2** and 50 cm³ of deionised water. Record your results, in a suitable form, in the space below.

	Must fa	Must fall in the range	
Mass of weighing bottle / g	4.08		
mass of weighing bottle + FA 2 / g	6.43	We can	
mass of weighing bottle + residual FA 2 / g	4.15 🔶	100% tra	
mass of FA 2 added / g	2.28	may be s	
initial temperature of water / °C	30.4	FA2 left	
lowest temperature reached / °C	28	FA2	
temperature fall, $\Delta T / °C$	-2.4		

We cannot assume 100% transfer as there may be some residual FA2 left in the weighing bottle and not added to FA2.

appropriate table of results

[1]

(ii) Use your results in **c(i)** to calculate a value for the enthalpy change of solution of NaHCO₃, ΔH_4 . Include the sign of ΔH_4 in your answer.

heat absorbed = mc ΔT = 50 × 4.18 × 2.4 = 502 J calculates energy change for vol of solution

 $M_{r} \text{ of NaHCO}_{3} = 23.0 + 1.0 + 12.0 + 3(16.0) = 84.0$ mol of NaHCO₃ = $\frac{2.28}{84.0} = 0.0271$ mol $\therefore \Delta H_{4} = + \frac{502}{0.0271} \text{ J mol}^{-1}$ = +18480 J mol^{-1} = +<u>18.5 kJ mol}^{-1} calculates ΔH_{4} , including sign</u>

enthalpy change, $\Delta H_4 = \dots + 18.5$ kJ mol⁻¹ [2]

(iii) Hence use your answer in **b(iii)** and **(c)(ii)**, determine the enthalpy change for the following reaction:



(d) Calculate the percentage error incurred when using the thermometer to measure the temperature change in **Experiment 1**.

% error incurred for temperature reading = $\left(\frac{2\times0.1}{6.0}\right) \times 100\% = \frac{3.33\%}{6.0}$	
As the difference between each reading on the thermometer is 0.2 °C, half the division is 0.1 °C. Working must clearly state 2×0.1 to show understanding that 2 temperature readings are taken (initial and final temperature).	[1] [Total: 18]

3 Investigation of reaction between propanone and iodine

 $\label{eq:FA3} \begin{array}{l} \textbf{FA 3} \text{ is } 1.00 \mbox{ mol } dm^{-3} \mbox{ sulfuric acid, } H_2SO_4. \\ \textbf{FA 4} \mbox{ is } 1.00 \mbox{ mol } dm^{-3} \mbox{ propanone, } CH_3COCH_3. \\ \textbf{FA 5} \mbox{ is an aqueous solution of iodine, } I_2. \\ \textbf{FA 6} \mbox{ is } 0.0100 \mbox{ mol } dm^{-3} \mbox{ sodium thiosulfate, } Na_2S_2O_3. \\ \textbf{FA 7} \mbox{ is } 0.50 \mbox{ mol } dm^{-3} \mbox{ sodium hydrogen carbonate, } NaHCO_3. \end{array}$

You are also provided with starch indicator.

The reaction between propanone and iodine in aqueous solution is shown in **reaction 1**. This reaction is first order with respect to both CH_3COCH_3 and H^+ ions.

reaction 1 $CH_3COCH_3(aq) + I_2(aq) \rightarrow CH_3COCH_2I(aq) + HI(aq)$

You are to investigate how the rate of reaction changes with $[I_2]$. To do this, you will prepare a reaction mixture containing **FA 3**, **FA 4** and **FA 5**. At timed intervals, you will withdraw five aliquots (portions) of the reaction mixture and quench the reaction with excess **FA 7**.

You will then titrate the remaining iodine in the resulting solutions against sodium thiosulfate (reaction 2).

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

Your titre values at the different times will indicate the concentration of I_2 remaining in the reaction mixture at those different times. Hence, the rate of reaction between CH₃COCH₃ and I_2 at the different times can be determined by graphical analysis of your results.

You should read all of the instructions on this page and the method on the next page before you start this experiment.

Recording your results

In an appropriate format in the space provided on **page 13** under the heading, **Results**, in **(b)**, prepare a table to record, for each of your aliquots, the

- transfer time in minutes and seconds,
- titration results (initial and final burette readings; and volume of **FA 6** added),
- time, which is the transfer time converted to minutes, to one decimal place (e.g. a transfer time of 2 min 27 s becomes 2 min + 27/60 min = 2.5 min).

Make certain that your recorded results show the precision of your working.

(a) Preparation and titration of the reaction mixture

Notes: You will perform each titration **once** only. Great care must be taken that you do not overshoot the end-point.

You should **keep the reaction mixture stoppered** except when removing aliquots. Once you have started the stopwatch, it must continue for the duration of the experiment. You must **not** stop it until you have finished this experiment.

You should aim **not** to exceed a maximum reaction time of 25 minutes for this experiment.

- 1. Using a 10 cm³ measuring cylinder, add about 10 cm³ of **FA 7** to each of the labelled boiling tubes, **1** to **5**.
- 2. Fill a burette with **FA 6**.
- 3. Use appropriate measuring cylinders to add to the conical flask labelled **reaction mixture**
 - 25.0 cm³ of **FA 3**,
 - 25.0 cm³ of **FA 4**.
- 4. Place 50.0 cm³ of **FA 5** into a 50 cm³ measuring cylinder.
- 5. Pour **FA 5** into the conical flask labelled **reaction mixture**. Start the stopwatch at the instant of mixing. **Insert the stopper** and swirl the mixture thoroughly to mix its contents.
- 6. At approximately <mark>4 minutes</mark>, transfer a 10.0 cm³ aliquot (portion) of the reaction mixture into the boiling tube labelled **1** using a pipette and shake the mixture. Read and record the transfer time (in minutes and seconds, to the nearest second) when half of the reaction mixture has emptied from the pipette.
- 7. Transfer all the contents of boiling tube **1** into a second conical flask. Rinse this boiling tube with deionised water and add the washings to the conical flask.
- Immediately titrate the I₂ in the second conical flask with FA 6 until the solution turns pale yellow. Then add about 1 cm³ of starch indicator. The solution will turn blue-black. Continue to titrate until the blue-black colour just disappears at the end-point. Record the final burette reading and the volume of FA 6 added.
- 9. Empty the contents of this conical flask into the waste bottle. Wash this conical flask thoroughly with water.
- 10. Repeat Step **6** to **9** four more times at about <mark>8 minutes, 12 minutes, 16 minutes and 20 minutes</mark>, transferring the aliquots into the boiling tubes labelled **2** to **5**.

(b) Results

Transfer time	Final burette reading /cm ³	Initial burette reading /cm ³	Volume of FA 6 added /cm ³	t /min
4 min 12 s	16.80	0.00	16.80	4.2
8 min 30 s	15.10	0.20	14.90	8.5
12 min 5 s	28.70	15.10	13.60	12.1
16 min 0 s	40.70	28.70	12.00	16.0
20 min 18 s	40.15	29.90	10.25	20.3

[5]

Units for seconds is 's', NOT 'sec'. WRONG headers include 'final volume' and 'initial volume'. Read the question and follow instructions. Transfer time must be included in your table, and t is in 1 dp.

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(c)(i) On the grid below, plot a graph of the volume of sodium thiosulfate, **FA 6**, on the y-axis, against time, t, on the x-axis.





(ii) Deduce the order of reaction with respect to the $[I_2]$ in reaction 1. Explain your answer.

Order Order of reaction is zero with respect to I₂

explanation Graph is a straight line with constant gradient / rate of reaction is

independent of $[I_2] / [I_2]$ decreases linearly with time / $[I_2]$ decreases

<u>at a constant rate</u>.

Clear reference to constant gradient and/or constant rate must be made.

(iii) Given that the reaction is first order with respect to both CH₃COCH₃ and H⁺ ions and using your answer from (c)(ii), write the rate equation for **reaction 1**.

Rate = k [CH₃COCH₃][H⁺] ecf

Question states 'This reaction is first order with respect to both CH₃COCH₃ and H⁺ ions.' So, rate equation MUST include CH₃COCH₃ and H⁺ ions.

(iv) The reaction between propanone and bromine proceeds by a similar mechanism as the reaction between propanone and iodine. How would the rate of reaction between propanone and bromine be compared with that of propanone and iodine? Explain your answer.

The rate would be the <u>same</u> as I_2 (and hence Br_2) does <u>not appear in the rate</u>

equation.

Since I_2 does not appear in the rate equation, Br_2 will also not appear in rate equation. The rate will not be affected by I_2 or Br_2 .

- (d) The initial rate of change of concentration of iodine, $[I_2]$, can be determined from the gradient of the graph at t = 0.0 min.
 - (i) Calculate the gradient of the graph at t = 0.0 min, showing clearly how you did this.

Gradient =
$$\frac{y_2 - y_1}{x_2 - x_1} = \frac{10.5 - 18}{19.6 - 1.2}$$

= <u>-0.408</u> cm³ min⁻¹ (3 s.f)

Show coordinates in workings; two chosen points should be at least 3 big squares apart in both directions

NEGATIVE sign must be included in the gradient. Make sure the formula of gradient is correctly applied.

Gradient = $cm^3 min^{-1}$ [1]

... [1]

(ii) Use your answer in (d)(i) to determine the rate of change of the amount of $S_2O_3^{2-}$ ions required in mol min⁻¹.

rate of change = (d)(i) × $\frac{0.010}{1000}$ = - 0.408 × $\frac{0.010}{1000}$ = - 4.08 × 10⁻⁶ mol min⁻¹ (3 s.f.)

Concentration of $S_2O_3^{2-}$ ions is 0.0100 mol dm⁻³.

Rate of change of the amount of $S_2O_3^{2-}$ required =mol min⁻¹[1]

(iii) Use your answer in d(ii) to determine change in amount of I_2 at t = 0.0 min in mol min⁻¹.

$$\begin{split} I_2 &\equiv 2S_2O_3^{2-} \\ \text{rate change of } I_2 &= \frac{1}{2} \times (d)(\text{ii}) \\ &= \frac{1}{2} \times -4.08 \times 10^{-6} \\ &= - \ \underline{2.04 \times 10^{-6}} \text{ mol min}^{-1} \text{ (3 s.f.)} \qquad \frac{1}{2} \times \text{c(ii)}] \end{split}$$

(iv) Using your answer in **d(iii)** and considering that a 10.0 cm³ aliquot (portion) of the reaction mixture was used, determine the rate of change of I_2 at t = 0.0 min in mol dm⁻³ min⁻¹

rate of change of $[I_2] = (d)(iii) \div \frac{10}{1000}$ divide by volume of 10cm³ aliquot = -2.04 x 10⁻⁶ ÷ $\frac{10}{1000}$ $\approx -2.04 x 10^{-4} \text{ mol dm}^{-3} \text{ min}^{-1} (3 \text{ s.f})$

Rate of change of $[I_2]$ at t = 0.0 min = - 2.04 x 10⁻⁴ mol dm⁻³ min⁻¹ [1]

(e) In Step 6, the aliquot is transferred into a boiling tube containing NaHCO₃. Explain why NaHCO₃ must be added and what effect failing to do it would have on the titre values.

NaHCO₃ must be added to react with the acid catalyst to <u>quench the reaction / acts</u>
<u>as a quenching agent</u>. If aliquots are not quenched, the titre values would be <u>lower</u>
[1]
<u>than expected</u>. [1]
[Total: 16]

If the reaction does not stop, the reaction will continue and more I_2 will be consumed. So, less I_2 will react with $S_2O_3^{2-}$, giving a lower expected titre value.

4 Planning: Measuring the activation energy for a reaction

Vitamin C, also known as ascorbic acid ($C_6H_8O_6$) is essential to health. However, in the presence of heat, ascorbic acid is easily oxidised to L-dehydroascorbic acid ($C_6H_6O_6$) as shown in **reaction 1** below and it no longer serves its purpose.

reaction 1 $C_6H_8O_6 \rightarrow C_6H_6O_6 + 2H^+ + 2e^-$

The rate equation for this reaction is

rate =
$$k [C_6 H_8 O_6]$$

where k is the rate constant.

The rate of oxidation of ascorbic acid can be determined by monitoring the amount of ascorbic acid left in the sample at various timed intervals. The oxidation of ascorbic acid becomes significant only at temperatures **40** °C and above.

Portions of the reaction mixture are

- removed at regular timed intervals,
- quenched by putting it in an ice bath,
- titrated against a standard solution of aqueous iodine, I2 as shown in reaction 2

reaction 2 $C_6H_8O_6 + I_2 \rightarrow C_6H_6O_6 + 2H^+ + 2I^-$

The activation energy, E_a , of the reaction can be determined from the equation.

$$\ln k = -\frac{E_a}{R} \left(\frac{1}{T}\right) + c$$

R is the molar gas constant.

T is the reaction temperature in kelvin.

k is the rate constant at a chosen temperature.

c is a constant for the reaction.

k can be determined from the initial rate of each experiment at different temperatures, *T*.

$$k = \frac{\text{Initial rate}}{[C_6H_8O_6] \text{ at t=0 min}}$$

(a) Plan an investigation, to determine the effect of temperature, T, on the rate of oxidation of ascorbic acid using the titrimetric method to monitor the rate as described above. The activation energy, E_a , is to be graphically determined.

Plotting ln *k* against $\frac{1}{\tau}$ gives a straight line of best fit. The gradient of this line is $-\frac{E_a}{R}$, where *R* is the molar gas constant.

You may assume that you are provided with

- five Vitamin C tablets (each tablet contains 500mg of ascorbic acid),
- deionised water,
- aqueous iodine, I₂,
- starch solution,
- ice,
- hot water bath,
- the equipment normally found in a school or college laboratory.

In order to prepare the reaction mixture at temperature, $T \,^{\circ}$ C, a total of 75 cm³ of deionised water is added to a conical flask. Ensure that the temperature of the deionised water has reached $T \,^{\circ}$ C before adding one Vitamin C tablet into the conical flask. The conical flask is then swirled to ensure the entire tablet has been dissolved.

Your plan should include brief details of:

- the reactants and conditions that you would use,
- the apparatus you would use,
- the procedure you would follow and the measurements you would make at different timings and temperatures,
- how you would determine the initial rate for each experiment by plotting an appropriate graph.

In your plan, it is **not** required to refer to concentrations or to perform calculations.

- 1. Fill a 50.00 cm³ burette with aqueous I₂.
- Using a second burette, add <u>75.0 cm³</u> of water into a 250 cm³ conical flask labelled 'Reaction'
-Mixture'... to use appropriate apparatus to measure volume
- 3. Place the conical flask of water in a water bath of 40 °C. Measure the temperature of the conical
- ••••• flask of deionised water using a <u>thermometer</u>. Once the flask of deionised water has reached 40 °C, add 1 Vitamin C tablet into the flask and swirl the flask to ensure all Vitamin C has been dissolved. Start the stopwatch once the tablet has been dissolved.
- After 5 minutes, use a <u>pipette</u> to transfer <u>10.0 cm³</u> aliquot of reaction mixture into a 250 cm³ <u>conical flask</u>.
- 5...Place the conical flask in the ice bath to quench the reaction.....
- 6. Add 5 drops of starch indicator into the conical flask and immediately titrate the reaction
-mixture with I2: The end point is reached when the solution in the conical flask turns from colourless to first permanent blue-black colour is seen. correct identification of end point
- 7. Repeat steps 4 to 6 and until a total of four aliquots have been titrated and their results
- recorded. Step 4 is repeated at appropriate timings of <u>10, 15, 20, and 25 minutes</u>. *need to withdraw at least 5 aliquots for titration at regular time intervals*
- 8...Repeat the experiment (from steps 2 to 7) for another four reaction mixtures at different temperatures (50 °C, 60 °C, 70 °C, 80 °C) by placing the 'Reaction Mixture' prepared in step 2
- in a water bath at the chosen temperature: *repeat experiment using water bath at different temperatures (at least 5); appropriate temperatures (> 40°C)*

appropriate apparatus used correct procedure (steps 3 to 5)

9. To determine the initial rate for each experiment

For each experiment, plot a graph of the <u>volume of I_2 </u> added on the y-axis <u>against time</u> on the xaxis. [Turn over

The initial rate is giver 2900 the gradient of the tangent and the course at t = 0 min.

This question was poorly attempted. Read the question very carefully and answer every bullet point that was asked.

- Inappropriate/Missing apparatus were used in the experiment. Include the capacity of the apparatus when used.
- Preparation of the reaction mixture was given in the question. You need to include it and add in necessary details (state the temperature, apparatus to measure the deionised water and when to start the stop watch).
- As shown in reaction 2, to determine how much of Vitamin C is left in the reaction mixture, Vitamin C must be titrated against I₂. So, I₂ must be in the burette. The end point is when there is NO MORE Vitamin C to react with I₂; So, the first dop of I2 will be detected by the blue black colour of starch. Starch must be added BEFORE the titration start.
- To plot a graph to get Volume of I_2 against time, 5 points must be plotted. So, we need to have 5 titration readings (ie 5 different timings).
- By finding out the gradient of the tangent at t=0 min for the graph of Volume of I_2 against time (reactant-time graph), this will give us the initial rate (k values) for each temperature.
 - [8]
- (b) Sketch the graph you would expect to obtain from 4(a) on the axes below.



[1]

(c) Describe how you would use your graph to determine the value of E_{a} .

From the gradient of the graph, $\frac{-E_a}{R}$ can be determined.	
gradient of straight-line graph = $\left(\frac{y_1 - y_2}{x_1 - x_2}\right) = -\frac{E_a}{R}$	
$\therefore E_a = -\left(\frac{y_1 \cdot y_2}{x_1 \cdot x_2}\right) \times 8.31 \text{ J mol}^{-1}$	[1]
	[Total: 10]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

action	reaction with	
cation	NaOH(aq)	NH₃(aq)
aluminium, A <i>l</i> ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH₄⁺(aq)	ammonia produced on heating	_
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe²+(aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg ²⁺ (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn ²⁺ (aq)	off-white ppt. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn²+(aq)	white ppt. soluble in excess	white ppt. soluble in excess

(b) Reactions of aqueous anions

ion	reaction
carbonate, CO3 ²⁻	CO ₂ liberated by dilute acids
chloride, C <i>l</i> ⁻ (aq)	gives white ppt. with Ag⁺(aq) (soluble in NH₃(aq))
bromide, Br⁻(aq)	gives pale cream ppt. with Ag ⁺ (aq) (partially soluble in NH ₃ (aq))
iodide, I⁻(aq)	gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq))
nitrate, NO₃⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil
nitrite, NO₂⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)
sulfate, SO₄²⁻(aq)	gives white ppt. with Ba ²⁺ (aq) (insoluble in excess dilute strong acids)
sulfite, SO ₃ ²-(aq)	SO ₂ liberated with dilute acids; gives white ppt. with Ba ²⁺ (aq) (soluble in dilute strong acids)

(c) Tests for gases

gas	test and test result	
ammonia, NH₃	turns damp red litmus paper blue	
carbon dioxide, CO ₂	gives a white ppt. with limewater (ppt. dissolves with excess CO ₂)	
chlorine, Cl ₂	bleaches damp litmus paper	
hydrogen, H ₂	"pops" with a lighted splint	
oxygen, O ₂	relights a glowing splint	
sulfur dioxide, SO ₂	turns aqueous acidified potassium manganate(VII) from purple to colourless	

(d) Colour of halogens

halogen	colour of element	colour in aqueous solution	colour in hexane
chlorine, Cl ₂	greenish yellow gas	pale yellow	pale yellow
bromine, Br2	reddish brown gas / liquid	orange	orange-red
iodine, I2	black solid / purple gas	brown	purple