EUNOIA JUNIOR COLLEGE
JC2 Preliminary Examination 2024
General Certificate of Education Advanced Level
Higher 2

CANDIDATE NAME	

CIVICS GROUP	2	3	–			INDEX NUMBER		

CHEMISTRY

9729/04

Paper 4 Practical

15 August 2024

2 hour 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidental Instructions

READ THESE INSTRUCTIONS FIRST

Write your name, civics group and registration number on the work you hand in.
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 paper clips, highlighters, 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.

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.

Shift
Laboratory

For Examiner's Use	
1	**/ 9**
2	**/11**
3	**/22**
4	**/13**
Total	**/55**

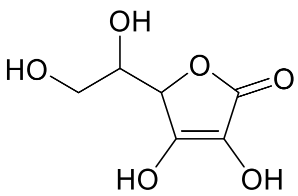
This document consists of **19** printed pages and **1** blank page.

Answer **all** the questions in the spaces provided.

For Examiner's Use

1 Investigation of some reactions of ascorbic acid

Ascorbic acid, more commonly known as vitamin C, is a water-soluble vitamin with molecular formula, $C_6H_8O_6$. Ascorbic acid is a well-known antioxidant that is reasonably stable in the solid form but oxidised quite rapidly by oxygen in air once dissolved in water.



ascorbic acid

FA 1 is a solution of ascorbic acid, $C_6H_8O_6$.
FA 2 is a solution of copper(II) sulfate, $CuSO_4$
FA 6 is 1.0 mol dm^{-3} hydrochloric acid, HCl

Carry out the following test. Carefully record your observations in Tables 1.1 and 1.2.

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

Table 1.1

	test	observations
(a)	Add 2 cm depth of aqueous silver nitrate to a clean dry boiling tube. Add 1 cm depth of aqueous sodium hydroxide slowly to the same tube.	
	Add aqueous ammonia slowly, with shaking until the precipitate just dissolves. You may use a clean glass rod to help dissolve the precipitate.	
	Add 1 cm depth of **FA 1** to this mixture and shake the tube. Place the boiling tube in the test-tube rack and leave it for 3 minutes.	
	Important: After about 3 minutes, pour the mixture down the sink and wash out the boiling tube several times with tap water.	

[2]

For Examiner's Use

Table 1.2

		test	observations
(b)	**(i)**	Add 2 cm depth of **FA 2** to a clean dry boiling tube. Add 1 cm depth of **FA 1** to the same tube and shake the tube. Gently heat the boiling tube until the liquid boils. Place the boiling tube in the test-tube rack and leave it to stand.	
	(ii)	Add 2 cm depth of **FA 2** to a clean dry boiling tube. Add 10 drops of **FA 6** to the same tube. Add 1 cm depth of **FA 1** to the same tube and shake the tube. Gently heat the boiling tube until the liquid boils. Place the boiling tube in the test-tube rack and leave it to stand.	

[3]

(c) In **(a)**, an organic product with molecular formula $C_6H_6O_6$ is obtained from ascorbic acid.

(i) Name the type of reaction that ascorbic acid undergoes in **(a)**.

.... [1]

(ii) State and explain the chemical change the **reagent** undergoes during the reaction in **(a)**.

....

.... [1]

For Examiner's Use

(iii) Explain why ascorbic acid is not expected to react with the reagent in **(a)**.

....

....

.... [1]

(d) The Cu^{2+} in **(b)** undergoes the same chemical change identified in **(c)(ii)**.

From the appearance of the copper-containing product, state

- the oxidation state of copper in the copper-containing product in **(b)(i)** ....
- the oxidation state of copper in the copper-containing product in **(b)(ii)** ....

[1]

[Total : 9]

BLANK PAGE

For Examiner's Use

2 Determination of the percentage by mass of ascorbic acid in a tablet

As ascorbic acid is readily oxidised, it is easier to analyse ascorbic acid using a redox titration rather than an acid-base titration.

When iodate ions, IO_3^-, are added to an acidic solution containing iodide ions, I^-, a redox reaction occurs to produce iodine, I_2.

equation 1 $$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$

The I_2 formed by this reaction is able to oxidise ascorbic acid to dehydroascorbic acid.

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

Due to this reaction the I_2 formed is immediately reduced to I^- as long as there is any ascorbic acid present. Once all the ascorbic acid has reacted, the excess I_2 is free to react with the starch indicator, forming the dark blue starch-iodine complex. This is the end-point of the titration.

In **2(a)**, you will perform titrations to determine the percentage by mass of ascorbic acid present in a vitamin C tablet.

FA 3 is a powdered vitamin C tablet, $C_6H_8O_6$
FA 4 is 0.0020 mol dm^{-3} potassium iodate, KIO_3
FA 5 is 0.200 mol dm^{-3} potassium iodide, KI
FA 6 is 1.0 mol dm^{-3} hydrochloric acid, HCl
Solution S is starch indicator

(a) (i) Preparation of standard solution of FA 3

1. Weigh the capped bottle containing solid **FA 3**. Record the mass in Table 2.1 on page 7.
2. Transfer all the solid **FA 3*** into a 250 cm^3 beaker.
3. Reweigh the empty capped container. Record this mass in Table 2.1 on page 7.
4. Dissolve the solid in about 70 cm^3 of deionised water.
5. Transfer all the solution into a 250 cm^3 volumetric flask.
6. Make up the solution to 250 cm^3 with deionised water and mix thoroughly. You need **FA 3 solution** for use in **3(a)** as well. Do not pour away after the titration.

* Gently tap the capped bottle on the benchtop to loosen the solid in the bottle before transfer and ensure that as much of solid **FA 3** is transferred into the beaker as possible without using any other aids.

For Examiner's Use

Table 2.1

mass of capped container and solid **FA 3** / g	
mass of emptied capped container / g	

(ii) Titration of FA 3 solution against FA 4

1. Fill the burette with **FA 4.**
2. Use the pipette to transfer 25.0 cm^3 of **FA 3 solution** into a 250 cm^3 conical flask.
3. Use a measuring cylinder to add 15.0 cm^3 of **FA 5** to the conical flask.
4. Use a measuring cylinder to add 5.0 cm^3 of **FA 6** to the conical flask.
5. Use a measuring cylinder to add 5.0 cm^3 of **solution S** to the conical flask.
6. Run **FA 4** from the burette into the flask. The end-point is reached when the **first permanent** trace of a dark blue colour is seen.
7. Record your titration results, to an appropriate level of precision, in Table 2.2.
8. Repeat points 2 to 8 until consistent titre values are obtained.

Table 2.2

final burette reading / cm^3					
initial burette reading / cm^3					
volume of **FA 4** used / cm^3					

[2]

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

volume of **FA 4** = ... cm^3 [3]

For Examiner's Use

(b) **(i)** Calculate the amount of IO_3^- ions present in the volume of **FA 4** calculated in **(a)(iii)**.

amount of IO_3^- ions =.. [1]

(ii) Calculate the amount of I_2 formed from the amount of IO_3^- ions in **(b)(i)**.

amount of I_2 formed =.. [1]

(iii) Calculate the concentration of ascorbic acid in **FA 3 solution**.

[ascorbic acid] in **FA 3 solution** =.. [2]

(iv) Hence, calculate the percentage by mass of ascorbic acid, $C_6H_8O_6$, in the tablet.
[A_r: H, 1.0; C, 12.0; O, 16.0]

percentage by mass of ascorbic acid in tablet = ... [2]

[Total: 11]

For Examiner's Use

3 Determination of the kinetics of the reaction between peroxodisulfate and iodide ions

FA 3 solution is the standard solution of vitamin C tablet prepared in **2(a)(i)**
FA 5 is 0.200 mol dm^{-3} potassium iodide, KI
FA 7 is 0.100 mol dm^{-3} ammonium peroxodisulfate, $(NH_4)_2S_2O_8$
Solution S is starch indicator

Peroxodisulfate ions, $S_2O_8^{2-}$, in **FA 7** oxidise iodide ions, I^-, in **FA 5** as shown below.

equation 3 $$S_2O_8^{2-}(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2SO_4^{2-}(aq)$$

The iodine, I_2, produced in equation 3 reacts immediately with ascorbic acid, from **FA 3**, as shown in equation 2.

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

Once all the ascorbic acid have reacted, the concentration of iodine rapidly increases and, due to the presence of starch in the reaction mixture, the dark blue colouration of the starch-iodine complex is formed.

The reaction in equation 3 is first order with respect to the iodide ion concentration, $[I^-]$.

You are to perform a series of experiments to determine the rate order for the reaction in equation 3 with respect to the peroxodisulfate ion concentration, $[S_2O_8^{2-}]$.

(a) Experiments

You will attempt five experiments.

- In **Experiment 1**, **Solution A** will be prepared as described on page 10.
- In the remaining experiments you will repeat the procedure from **Experiment 1**, but using volumes of **FA 7** of your choice.

For each experiment, you note the time taken, ***t***, for the solution to turn dark blue.

For Examiner's Use

(i) Experiment 1

Fill the burette labelled **E3** with **FA 7**.

- Transfer 25.00 cm^3 of **FA 7** to a conical flask.

Solution A

- Using a 10 cm^3 measuring cylinder, add 5.0 cm^3 of **FA 3 solution** to the beaker labelled **Solution A**.
- Using a 25 cm^3 measuring cylinder, add 20.0 cm^3 of **FA 5** to the same beaker.
- Using a 10 cm^3 measuring cylinder, add 5.0 cm^3 of **solution S** to the same beaker.
- Mix the contents thoroughly by swirling the beaker.

1. Pour **Solution A** rapidly into the conical flask containing **FA 7**. Start the stopwatch when you have added about half of **Solution A**.
2. Mix the contents thoroughly by swirling the flask.
3. Stop the stopwatch when the dark blue colour first appears.
4. Note the time elapsed, ***t***, to the nearest second.
5. Wash the conical flask and beaker thoroughly with water and allow to drain.

(ii) Experiments 2 to 5

You are now to perform **four** other experiments in order to determine the rate order with respect to $[S_2O_8^{2-}]$ for equation 3. You should number these experiments **2** to **5**.

In each experiment, the volumes of **FA 3**, **FA 5** and **solution S** are the same as those used in **Experiment 1**.

Select suitable volumes of **FA 7**, $V_{\textbf{FA 7}}$, ensuring that your chosen volumes:

- allow you to obtain sufficient data to determine the order through the plotting of a graph,
- are not larger than the volume used in **Experiment 1**,
- are not less than 15.00 cm^3.

In each case, the total volume of the reaction mixture must be kept the same as that used in **Experiment 1**, by adding deionised water as required.

For Examiner's Use

(b) Results

The volumes of **FA 3**, **FA 5** and **solution S** are not changed in these experiments, and do not need to be recorded.

Prepare a table in the space provided below in which to record, for each experiment:

- all volumes apart from those of **FA 3**, **FA 5** and **solution S**,
- the value of ***t***,
- calculated values for the experimental rate of reaction.

Record your results in the table.

(i) Table of results

[5]

(ii) Use your answer to **2(b)(iii)**, calculate the amount of ascorbic acid used in each experiment in **3(a)**.

amount of ascorbic acid =.. [1]

(iii) Use your answer to **3(b)(ii)**, and the equations for the reactions involved, to calculate the amount of peroxodisulfate ions, $S_2O_8^{2-}$, that reacted in each experiment in **3(a)**.

amount of $S_2O_8^{2-}$ =.. [1]

For Examiner's Use

(iv) Use your answer to **3(b)(iii)** to calculate the change in concentration of $S_2O_8^{2-}$, $[S_2O_8^{2-}]$, that occurred when enough iodine was produced to produce the dark blue colour in each experiment in **3(a)**.

change in $[S_2O_8^{2-}]$ = ... [1]

(v) The expression below shows the experimental rate of this reaction as the change in concentration of $S_2O_8^{2-}$ per unit time.

$$\text{experimental rate} = -\frac{\text{change in } \left[S_2O_8^{2-}\right]}{\text{time, } \boldsymbol{t}} \times 10^6 \ \mu\text{mol dm}^{-3} \ \text{s}^{-1}$$

(1 μmol = 10^{-6} mol)

Complete your table on page 11 by calculating the experimental rates of reaction for all 5 experiments, taking into consideration the units.

If you are unable to calculate a value for the change in $[S_2O_8^{2-}]$ in **3(b)(iv)**, use the value -2.50×10^{-4} mol dm^{-3}. (Note: this is not the actual value.) [5]

(c) Plot a graph of experimental rate on the *y*-axis against $V_{\textbf{FA 7}}$ on the *x*-axis.

The scales of both axes must be chosen to provide an origin.

Draw the best-fit straight line through the origin, taking into account all of your plotted points.

[3]

(d) By considering the shape of the graph in **(c)**, state and explain the order of the reaction with respect to $[S_2O_8^{2-}]$.

....

....

....

.... [2]

For Examiner's Use

(e) The order of reaction with respect to [I^-] is one. With reference to experiment **1**, state and explain the expected time taken for the appearance of the dark blue colour when the experiment is carried out using a mixture comprising the following:

- 10.0 cm^3 of **FA 3**
- 10.0 cm^3 of **FA 5**
- 25.00 cm^3 of **FA 7**
- 5.0 cm^3 of deionised water
- 5 cm^3 of **solution S**

....

....

....

....

....

....

.... [2]

(f) A student suggested that a more accurate timing can be obtained when the volume of **FA 3 solution** is measured with a burette rather than a measuring cylinder.

A teacher said that the student's claim is incorrect. Explain why this is so.

....

....

....

.... [2]

[Total: 22]

4 Planning

For Examiner's Use

Ascorbic acid is a weak monobasic acid with a pK_a of 4.17 at 25 ºC. Besides acid-base and redox titration, the concentration of ascorbic acid in a standard solution of **FA 3** prepared in **2(a)(i)** can also be determined by thermometrically.

This involves performing a series of experiments using different volumes of aqueous sodium hydroxide and the standard solution of **FA 3** which together give a total volume of 50 cm^3. The temperature change, ΔT, for each experiment will be determined and a graph of ΔT against the volume of NaOH used will then be plotted.

The volume of NaOH, V_{neut}, which gives the maximum temperature change, ΔT_{max}, are obtained from the graph.

V_{neut} can be used to calculate the concentration of ascorbic acid in the standard solution.

ΔT_{max} can be used to calculate the heat change, q, for this experiment. Using q, a value for the the enthalpy change of neutralisation, ΔH_{neut}, of vitamin C can be determined simultaneously.

(a) Plan an investigation to determine the concentration of vitamin C in a standard solution of **FA 3**, as well as the enthalpy change of neutralisation of ascorbic acid, thermometrically.

You may assume that you are provided with:

- a standard solution of **FA 3**,
- 0.100 mol dm^{-3} sodium hydroxide, NaOH,
- The equipment normally found in a school or college laboratory.

In your plan you should include brief details of:

- the apparatus you would use,
- the procedure you would follow,
- the measurements you would take,
- how the data measured would be used to determine values needed for the plotting of the graph.

....

....

....

....

....

....

For Examiner's Use

.... [5]

For Examiner's Use

(b) (i) Sketch the graph that you would expect to obtain on the axes in Fig. 4.1.

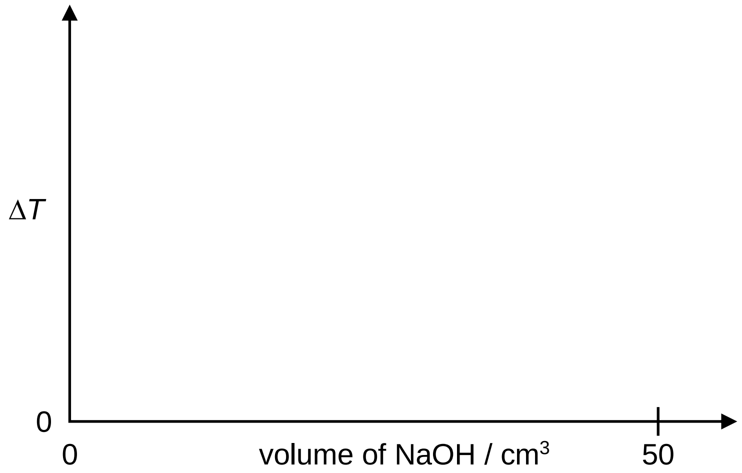


Fig. 4.1

[2]

(ii) Explain how the maximum temperature change, ΔT_{max}. and the corresponding volume of NaOH, V_{neut}, can be determined. You may find it useful to show how this might be done on your graph in **4(b)(i)**.

....

....

....

....

....

.... [2]

(c) (i) Derive an expression for the concentration of ascorbic acid in the standard solution of **FA 3**, in terms of V_{neut} only.

....

....

....

....

.... [2]

(ii) Derive an expression for the enthalpy change of neutralisation of ascorbic acid, in terms of ΔT_{max} and V_{neut} only.

For Examiner's Use

Assume that the specific heat capacity of the solution is 4.2 J cm^{-3} K^{-1}.

....

....

....

....

.... [2]

[Total: 13]

Qualitative Analysis Notes
[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	*reaction with*	
	NaOH(aq)	NH_3(aq)
aluminium, Al^{3+}(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH_4^+(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), Fe^{2+}(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, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

(b) Reactions of anions

anion	***reaction***
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $Cl^-(aq)$	gives white ppt. with $Ag^+(aq)$ (soluble in $NH_3(aq)$)
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 $NH_3(aq)$)
nitrate, $NO_3^-(aq)$	NH_3 liberated on heating with $OH^-(aq)$ and Al foil
nitrite, $NO_2^-(aq)$	NH_3 liberated on heating with $OH^-(aq)$ and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, $SO_4^{2-}(aq)$	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, NH_3	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, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I_2	black solid / purple gas	brown	purple