VICTORIA JUNIOR COLLEGE
PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME ……………………………………………………………..

CT GROUP ……………………………..

CHEMISTRY

9729/04

Paper 4 Practical

24 Aug 2023

Candidates answer on the Question Paper.

2 hours 30 minutes

Additional Materials: As listed in the instructions below

READ THESE INSTRUCTIONS FIRST

Write your name and CT group on all 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 a 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 **17** and **18**.

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	
2	
3	
4	
Total	**/ 55**

This document consists of **18** printed pages and **2** blank pages.

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

1 Inorganic and organic qualitative analysis

In this question, you will carry out tests on two solutions, **FA 1** and **FA 3**, and make conclusions about them.

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

Test and identity any gases evolved.

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

(a) **(i)** **FA 1** is an aqueous solution that contains two cations and one anion listed in the Qualitative Analysis notes.

You will carry out the tests in Table 1.1, which will allow you to identify the two cations present in **FA 1**.

Table 1.1

	tests	observations
1	Test the **FA 1** solution using Universal Indicator paper. Using the colour chart provided, record the pH of the solution.	
2	To 1 cm depth of **FA 1**, add 1 cm depth of KI. Let the solution stand for 1 min and observe any colour change that occurs. To the resulting solution, add 5 drops of starch solution.	
3	To 2 cm depth of **FA 1**, add aqueous ammonia dropwise with shaking till 4 cm depth of aqueous ammonia has been added. Swirl and filter the mixture, collecting the filtrate in a clean test-tube. The filtrate is **FA 2** which should be put to one side for use in **Test 4**.	

4	To 1 cm depth of **FA 2**, carefully add nitric acid dropwise until no further change is seen.	

[3]

(ii) Identify the two cations in **FA 1** and state the evidence for each cation by completing Table 1.2.

Table 1.2

cations	evidence

[2]

(iii) With the aid of a balanced equation, explain your observation in **Test 1** of **Table 1.1** above.

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.... [2]

(iv) One of the cations present in **FA 1** reacts with the KI solution added in **Test 2** of **Table 1.1** above.

Suggest a balanced equation to represent this reaction.

.... [1]

(v) Only one anion is present in **FA 1**. The anion is not carbonate or sulfite and does not contain any nitrogen.

Describe and carry out a series of tests that will allow you to identify the anion.

Before carrying out the tests, you are required to dilute the given **FA 1** solution by adding 1 cm depth of **FA 1** solution to a clean test-tube, followed by adding deionised water until the test-tube is half full.

Use the diluted solution to carry out your proposed tests.

[2]

(vi) Use your results in **(a)(v)** to identity the anion present in **FA 1**.

Anion present: ....

[1]

(b) **(i)** **FA 3** is an aqueous solution containing an organic compound with only one functional group present.

Perform **Test 1** in Table 1.3. There is no need to perform **Test 2** as the observation has been completed for you.

Table 1.3

	tests	observations
1	Add about 1 cm depth of **FA 3** in a test-tube. To this test-tube, add 6 drops of sodium hydroxide solution, followed by adding iodine solution, dropwise, until a permanent orange/red colour is present. Half fill a 250 cm^3 beaker with warm water. Immerse the test-tube into the warm water for two minutes.	
2	Add about 1 cm depth of **FA 3** in a test–tube. To this test–tube, add 2 drops of potassium dichromate (VI), followed by 1 cm depth of dilute sulfuric acid. Half fill a 250 cm^3 beaker with warm water. Immerse the test-tube into the warm water for two minutes.	No observable change

[1]

(ii) State the functional group present in **FA 3** and support your answer with evidence from Table 1.3.

functional group	evidence

[2]

[Total: 14]

2 Determination of the value for the number of water of crystallisation, *n*, in an iron(II) salt, $FeSO_4 \cdot nH_2O$

FA 4 is hydrated iron(II) sulfate, $FeSO_4 \cdot nH_2O$.
FA 5 is 1.00 mol dm^{-3} dilute sulfuric acid, H_2SO_4.
FA 6 is 0.0100 mol dm^{-3} potassium manganate(VII), $KMnO_4$.

You are to carry out titration to determine the number of water of crystallisation, *n*, in **FA 4**, which is the hydrated iron(II) sulfate, $FeSO_4 \cdot nH_2O$.

This involves preparing a standard solution **FA 7** by dissolving a fixed mass of **FA 4** in dilute sulfuric acid, **FA 5** with the use of a volumetric flask. **FA 6** is then titrated with **FA 7**.

The two half–equations for the titration are as follows:

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

(a) Procedure

1. Weigh the **capped** container containing solid **FA 4**. Record the mass in your table in the space provided on Page 6.
2. Transfer all the solid **FA 4** into a 150 cm^3 beaker.
3. Reweigh the empty **capped** container. Record this mass in your table on Page 7.
4. Using a 50 cm^3 measuring cylinder, add 50.0 cm^3 of **FA 5** to dissolve the solid **FA 4** in the beaker. Stir carefully and allow time for the entire amount of solid to dissolve.
5. Quantitatively transfer the resultant solution in the beaker into a 250 cm^3 volumetric flask. Make up to the mark with distilled water. Shake to obtain a homogeneous solution. Label this solution **FA 7**.
6. Fill a burette with **FA 6**.
7. Pipette 25.0 cm^3 of **FA 7** into a 250 cm^3 conical flask.
8. Use the 50 cm^3 measuring cylinder to add 25.0 cm^3 of **FA 5** into the conical flask.
9. Run **FA 6** from the burette into the conical flask. The end-point is reached when a visible colour change is observed.
10. Record your titration results, to an appropriate level of precision, in the space provided on Page 6.
11. Repeat steps 7 to 10 until consistent results are obtained.

Results

[5]

(b) **(i)** From your titration results in **(a)**, obtain a suitable volume of **FA 6** to be used in your calculation. Show clearly how you obtained this volume.

average volume of **FA 6** used = …………………… cm^3 [1]

(ii) Calculate the amount of MnO_4^- ions present in the volume of **FA 6** calculated in **(b)(i)**.

amount of MnO_4^- used = …………………… mol [1]

(iii) Calculate the amount of Fe^{2+} ions present in 25.0 cm^3 of **FA 7**.

amount of Fe^{2+} in 25.0 cm^3 of **FA 7** = ………………… mol [1]

(iv) Calculate the amount of Fe^{2+} ions present in 250 cm^3 of **FA 7**.

amount of Fe^{2+} ions in 250 cm^3 of **FA 7** = ………………… mol [1]

(v) Calculate the M_r of the hydrated iron(II) sulfate, $FeSO_4{\bullet}nH_2O$, in your weighed sample and hence the value of *n*.
[A_r: Fe, 55.8; S, 32.1; O, 16.0; H, 1.0]

M_r of the hydrated iron(II) sulfate = ………………………..

n = ……..…….

[3]

(c) **(i)** Iron(II) sulfate in solution is readily oxidised by air to form iron(III) sulfate.

State the effect on M_r of the hydrated iron(II) sulfate and the value of *n* calculated in **(b)(v)**, if some of your sample of **FA 4** had oxidised before you carried out the titration. Explain your answer.

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[2]

(ii) With the help of relevant calculations, show that the iron(II) is less stable in air under alkaline conditions compared to acidic conditions.

$Fe^{3+} + e^- \rightleftharpoons Fe^{2+}$ $E^\ominus = +0.77$ V
$Fe(OH)_3 + e^- \rightleftharpoons Fe(OH)_2 + OH^-$ $E^\ominus = –0.56$ V
$O_2 + 4H^+ + 4e^- \rightleftharpoons 2H_2O$ $E^\ominus = +1.23$ V
$O_2 + 2H_2O + 4e^- \rightleftharpoons 4OH^-$ $E^\ominus = +0.40$ V

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[2]

[Total: 16]

3 Investigation of the kinetics of the acid-catalysed iodination of propanone reaction

FA 5 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4
FA 8 is 1.00 mol dm^{-3} propanone, CH_3COCH_3
FA 9 is an aqueous solution of iodine, I_2
FA 10 is 0.0100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$
Solid sodium hydrogencarbonate

You are also provided with a starch indicator.

Iodine and propanone react together in aqueous solution according to the equation with acid as a catalyst.

$$CH_3COCH_3\,(aq) + I_2\,(aq) \xrightarrow{H^+} CH_3COCH_2I\,(aq) + HI\,(aq)$$

You are to investigate the order of reaction with respect to I_2.

A reaction mixture containing **FA 5**, **FA 8**, and **FA 9** is first prepared. At different chosen times, aliquots (fixed volumes) of this reaction mixture are removed and quenched using solid sodium hydrogencarbonate**.**

The remaining amount of I_2 at different times can then be determined by titration against **FA 10**.

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

The required order of reaction can be obtained by the graphical analysis of your results.

(a) (i) 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.

Once you have started the stopwatch, it must continue running for the duration of the experiment. You must **not** stop it until you have finished the experiment.

You should aim **not** to exceed a maximum reaction time of **20** minutes for this experiment.

In an appropriate format in the space provided, prepare a table in which to record for each aliquot

- the time of transfer, t, in minutes and seconds,
- the decimal time, t_d, to 0.1 min, for example, if t = 4 min 33 s then t_d = 4 min + 33/60 min = 4.6 min
- the burette readings and the volume of **FA 10** added.

Safety:
Propanone is flammable. Transfer your titrated solutions into the waste bottle for later disposal. Keep this bottle stoppered when not in use.

Keep the conical flask stoppered except when removing aliquots.

1. Fill a burette with **FA 10**.

2. Using a 25 cm^3 measuring cylinder, add 25.0 cm^3 of **FA 5** to a conical flask.
3. Using another 25 cm^3 measuring cylinder, add 25.0 cm^3 of **FA 8** to the same conical flask.
4. Using a 50 cm^3 measuring cylinder, add 50.0 cm^3 of **FA 9** to the same conical flask. Start the stopwatch, **insert the stopper** and swirl the mixture thoroughly to mix its contents.
5. Use a pipette to withdraw 10.0 cm^3 of the mixture and transfer it to another conical flask. Replace the stopper in the flask containing the original reaction mixture.
6. At approximately 2 minutes, add 1g (~one spatula full) of sodium hydrogencarbonate to the conical flask containing 10.0 cm^3 portion and shake to mix.
7. Titrate the iodine in this solution with **FA 10**. Add about 1 cm^3 of starch indicator when the colour of the solution turns pale yellow. The solution will turn blue–black. The end–point is reached when the blue–black colour just disappears. Record your results.
8. Empty the contents of this conical flask into the waste bottle. Wash this conical flask thoroughly with water.
9. Repeat steps **5** to **8** until a total of **five** aliquots have been titrated and their results recorded.

Results

[4]

(ii) On Fig 3.1, plot a graph of **volume of sodium thiosulfate, FA 10**, on the y–axis, against time, ***t***, on the x–axis. Start the x–axis at ***t*** = 0. You should choose a scale which will allow you to extrapolate your graph back to ***t*** = 0.

Draw the most appropriate best–fit line taking into account all of your plotted points.

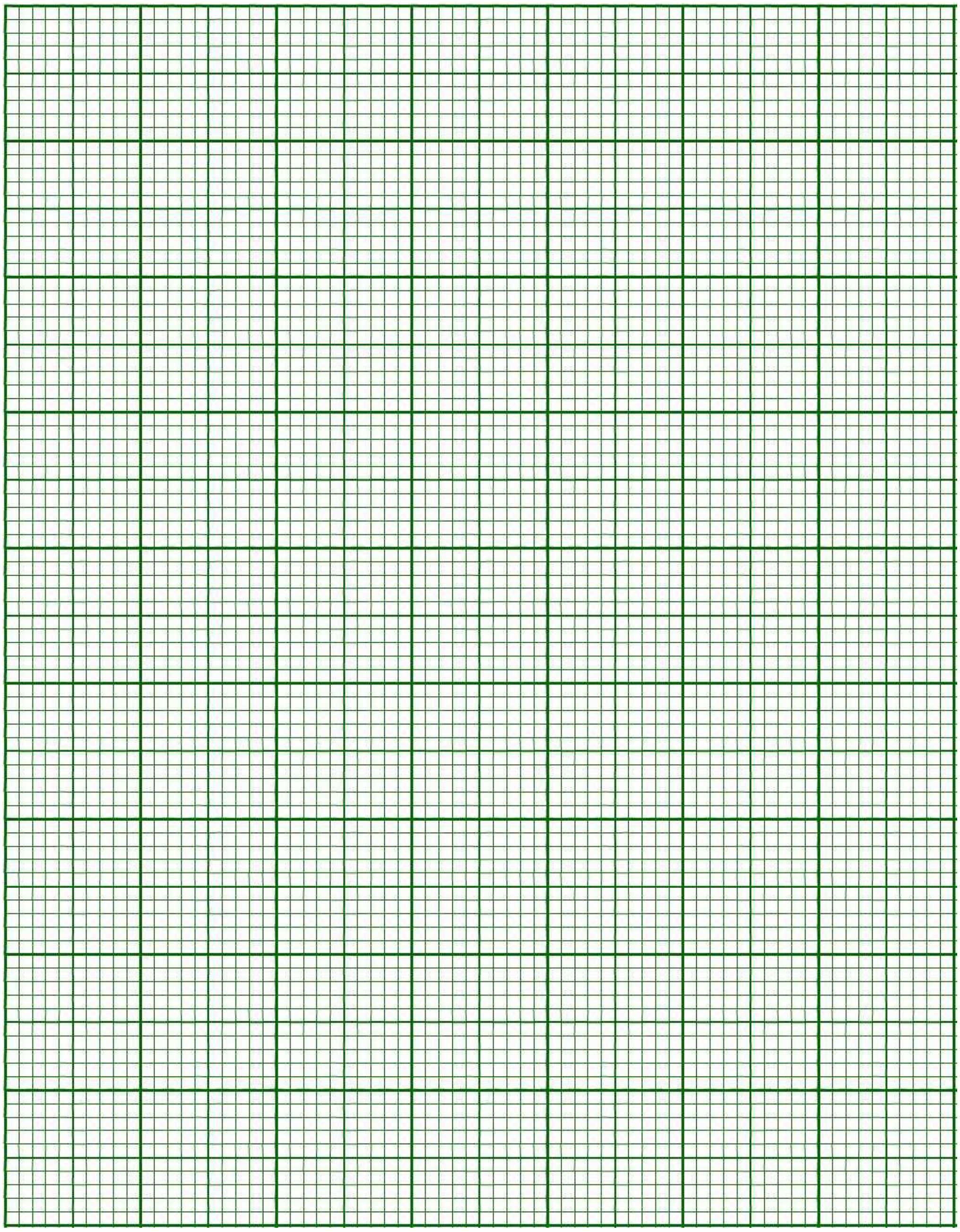

Fig 3.1

[3]

(iii) Deduce the order of reaction with respect to the I_2. Explain your answer.

order...................................

explanation..

....

[1]

(b) **(i)** Using the value obtained from the y-intercept from **Fig 3.1**, determine the concentration of iodine in **FA 9**.

[3]

(ii) Hence, suggest why concentration of propanone used in the experiment is much higher than that of iodine.

....

.... [1]

(c) This reaction is first order with respect to both CH_3COCH_3 and H^+ ions.

(i) Write the rate equation for the acid-catalysed iodination of propanone.

.... [1]

(ii) Sketch on Fig 3.2 the shape of the graph when the following changes are made. Include your original shape of the graph obtained in Fig 3.1.

A – $[H^+]$ is **doubled** while keeping the rest of the concentration of the reactants unchanged.

B – $[I_2]$ is **halved** while keeping the rest of the concentration of the reactants unchanged.

Fig 3.2

[2]

[Total: 15]

4 Planning: Determine the exact concentration of sodium hypochlorite in household bleach

Household bleach is typically 5% (w/v) aqueous sodium chlorate(I), NaC*l*O. The oxidation of propanone, CH_3COCH_3, by sodium chlorate(I), NaC*l*O, found in bleach, is known to be exothermic. The product chloroform, $CHCl_3$, produced is a possible **carcinogen** and it can vapourise easily at room temperature.

$$CH_3COCH_3 + 3NaClO \rightarrow CH_3COONa + CHCl_3 + 2NaOH$$

A series of experiments can be performed to determine the exact concentration of NaC*l*O in bleach. A fixed volume of NaC*l*O is to be mixed with different volumes of CH_3COCH_3 and distilled water such that the total volume is kept constant. Since the total volume of mixture remains the same, the temperature rise, ΔT, is a direct measure of the heat given out by the reaction. The maximum amount of heat is evolved when all the NaC*l*O present is completely reacted with CH_3COCH_3.

You may assume you are provided with:

- 50 cm^3 of 13.6 mol dm^{-3} of propanone
- 500 cm^3 of bleach of approximately 5% (w/v) aqueous sodium chlorate(I) (**corrosive** and **burns skin**)
- styrofoam cup
- thermometer
- the equipment normally found in a school or college laboratory.

(a) Given that 5% (w/v) sodium hypochlorite has a density of 1.093 g cm^{-3}, calculate the concentration of the sodium hypochlorite in mol dm^{-3}.

1% (w/v) means 1 gram of solute per 100 grams of solution.

[1]

(b) **(i)** Determine a suitable concentration of propanone such that 25 cm^3 of bleach will react completely with 25 cm^3 of the propanone. Justify your answer with calculations.

[2]

(ii) Describe how you would make a solution of propanone with the concentration determined in **(b)(i)**.

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[1]

(c) Plan an experiment to collect sufficient data to allow a graph of temperature rise against the volume of propanone to be drawn. On the graph, two best–fit lines are drawn. One line is drawn using data before the end–point and the second line using the remaining data. These lines are then extrapolated until they intersect.

In your plan you should use the solution of propanone you planned in **(b)(ii)** and the solutions provided.

In your plan you should include details of
- the apparatus you would use,
- the procedure you would follow,
- the measurements you would make,
- any safety precautions you would take.

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………………………………………………………………………………………………….. [4]

(d) Sketch on Fig 4.1, the graph you would expect to obtain from your results. Indicate clearly on your sketch how you would determine:

- $\boldsymbol{V}_{\mathrm{end}}$, the volume of CH_3COCH_3 needed to completely react with $\mathrm{NaC}l\mathrm{O}$,
- $\Delta\boldsymbol{T}_{\mathrm{max}}$, the maximum temperature rise when stoichiometric amount of CH_3COCH_3 and $\mathrm{NaC}l\mathrm{O}$ reacted.

Fig 4.1

[2]

[Total: 10]

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 → (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

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