



VICTORIA JUNIOR COLLEGE
PRELIMINARY EXAMINATION
Higher 2

CANDIDATE
NAME

CT GROUP

CHEMISTRY

9729/04

Paper 4 Practical

31 Aug 2020

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 15 and 16.

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	/ 12
2	/ 13
3	/ 17
4	/ 13
Total	/ 55

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

- 1 **X** is a solid sample of sodium ethanedioate, $\text{Na}_2\text{C}_2\text{O}_4$ and ethanedioic acid, $\text{H}_2\text{C}_2\text{O}_4$, a weak acid.

FA 1 is prepared by dissolving 2.00 g of **X** in a 250 cm^3 volumetric flask and making up to the mark with deionised water.

FA 2 is 0.0100 mol dm^{-3} sodium hydroxide, NaOH(aq) .

FA 3 is 0.0200 mol dm^{-3} potassium manganate(VII), $\text{KMnO}_4\text{(aq)}$.

Titration of **FA1** against **FA 2**

- (a)
1. Fill a burette with **FA 2**.
 2. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
 3. Add 2–3 drops of thymolphthalein indicator to the conical flask.
 4. Titrate **FA1 against FA 2** until a distinct colour change is observed.
 5. Carry out as many titrations as you deem necessary to obtain consistent results.
 6. Record in an appropriate form all of your burette readings and the volume of **FA 2** added in each titration.

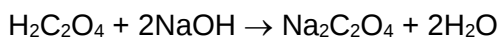
Results

[4]

- (b) (i) From your titration results, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

25.0 cm^3 of **FA 1** required cm^3 of **FA 1**. [1]

The equation for the reaction between sodium hydroxide and ethanedioic acid in this experiment is shown below:



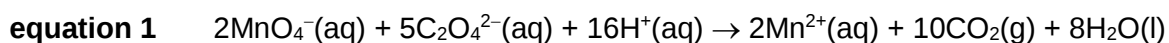
- (ii) Calculate the amount of ethanedioic acid in 25.0 cm^3 of **FA 1**.

amount of $\text{H}_2\text{C}_2\text{O}_4$ in 25.0 cm^3 of **FA 1** = mol [1]

- (c) When 25.0 cm³ of **FA 1** was titrated with **FA 3**, 25.05 cm³ of **FA 3** was required to reach the end-point.
- (i) Calculate the amount of potassium manganate(VII), **FA 3**, required to react with 25.0 cm³ of **FA 1**.

amount of KMnO₄ = mol [1]

- (ii) The equation for the reaction between acidified manganate(VII) ions and ethanedioate ions is shown in **equation 1** below.



Calculate the total amount of ethanedioate ions in 25.0 cm³ of **FA 1**.

total amount of C₂O₄²⁻ in 25.0 cm³ of **FA 1** = mol [1]

- (iii) Use your answers from (b)(ii) and (c)(ii) to calculate the amount of ethanedioate ions which came from the sodium ethanedioate in 25.0 cm³ of **FA 1**.

amount of C₂O₄²⁻ from Na₂C₂O₄ in 25.0 cm³ of **FA 1** = mol [1]

- (iv) Calculate the amount of sodium ethanedioate in **FA 1**.

amount of Na₂C₂O₄ in **FA 1** = mol [1]

- (v) Hence, calculate the percentage by mass of sodium ethanedioate in sample **X**.
 [A_r: Na, 23.0; C, 12.0; O, 16.0]

Percentage by mass of sodium ethanedioate in **X** =[2]

[Total: 12]

2 To study the kinetics of the Mn^{2+} catalysed reaction between MnO_4^- and $\text{C}_2\text{O}_4^{2-}$

FA 3 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), $\text{KMnO}_4(\text{aq})$.

FA 4 is $0.200 \text{ mol dm}^{-3}$ $\text{Na}_2\text{C}_2\text{O}_4(\text{aq})$.

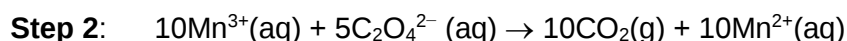
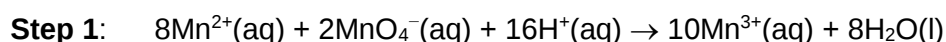
FA 5 is 2.00 mol dm^{-3} sulfuric acid, H_2SO_4 .

FA 6 is $0.200 \text{ mol dm}^{-3}$ $\text{MnSO}_4(\text{aq})$.

FA 7 is $0.400 \text{ mol dm}^{-3}$ $\text{KI}(\text{aq})$.

FA 8 is $0.00400 \text{ mol dm}^{-3}$ $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$.

In this experiment, you will determine the order of reaction with respect to MnO_4^- for the reaction between MnO_4^- and $\text{C}_2\text{O}_4^{2-}$, catalysed by Mn^{2+} ions. The mechanism for the catalysed reaction is shown by **step 1** and **step 2** below:



- (a) (i) With reference to the mechanism above and the uncatalysed reaction in **equation 1**, explain why the addition of Mn^{2+} will speed up the reaction.

.....

 [1]

Preparation and titration of the reaction mixture

Notes: Read through the instructions carefully before starting any practical work.

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 **15 minutes** for this experiment.

1. Fill a burette with **FA 8**.
2. Use a measuring cylinder to place the following in a 250 cm³ conical flask.
 - 50 cm³ of **FA 4**
 - 5 cm³ of **FA 5**
 - 15 cm³ of **FA 6**
 - 45 cm³ of deionised water
3. Use a measuring cylinder to add 50 cm³ of **FA 3** into the conical flask in step 2. Start the stopwatch simultaneously and swirl the mixture thoroughly to mix its contents. This is the reaction mixture.
4. Use a measuring cylinder to add 10 cm³ of **FA7** into a 100 cm³ conical flask.
5. Use a teat pipette (dropper) to withdraw a 10 cm³ aliquot (portion) of the reaction mixture and then transfer it into a 10 cm³ measuring cylinder.
6. **Immediately** pour this aliquot of the reaction mixture into the 100 cm³ conical flask containing **FA7**, and vigorously swirl the mixture. Read and record the time of transfer in minutes and seconds, to the nearest second, when the aliquot is added.
7. **Immediately** titrate the iodine formed in the 100 cm³ conical flask with **FA8** until the solution turns pale yellow. Add 10 drops of starch indicator to 100 cm³ conical flask and the solution will turn blue-black. Continue the titration until the blue-black colour just disappears.
8. Wash out the 100 cm³ conical flask with water.
9. Quickly repeat steps 4 to 8 until a total of **five** aliquots have been titrated.
10. In the space provided below, prepare a table and record the following for each aliquot:
 - the time of addition of **FA 7**, t , in minutes and seconds
 - the decimal time, t_d , in minutes, to 0.1 min, for example, if $t = 4 \text{ min } 33 \text{ s}$, then $t_d = 4 \text{ min} + 33/60 \text{ min} = 4.6 \text{ min}$
 - the burette readings and the volume of **FA 8** added

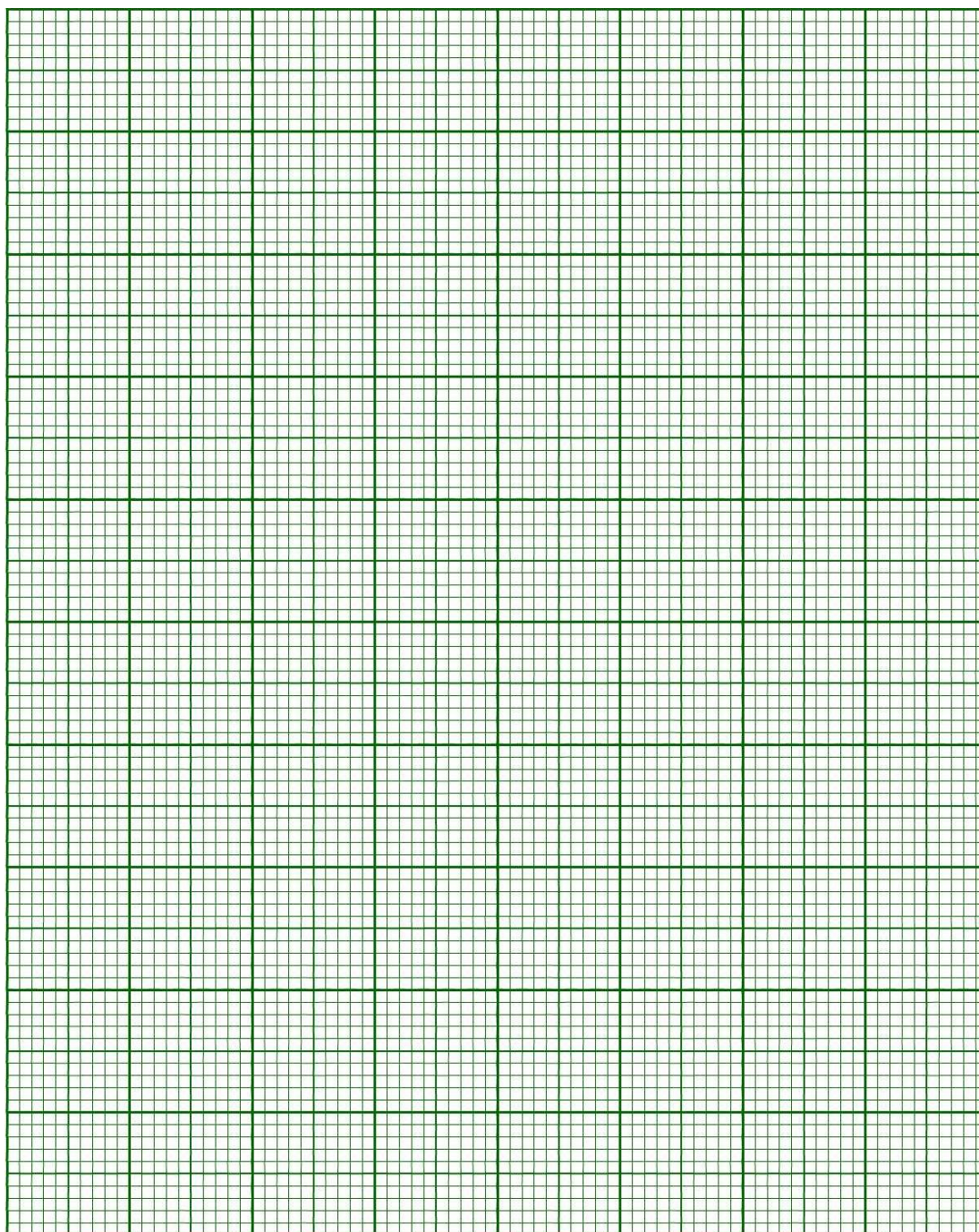
Results

(ii) Explain why a large excess of **FA 4** was added in step 2 of the procedure.

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.....
.....[1]

(iii) Plot a graph of the volume of **FA8** added on the y-axis, against decimal time, t_d , on the x-axis on the grid below.

Draw the most appropriate best-fit curve taking into account all of your plotted points.



[3]

During the experiment, **FA 7** was added to aliquots of the reaction mixture to stop the reaction. The reaction stops because **FA 7** reacts with all the MnO_4^- present in the aliquot. The I_2 evolved is then titrated with **FA 8**.

- (iv) State and explain the relationship between the concentration of MnO_4^- and volume of **FA 8** in this experiment.

.....

 [1]

- (v) It has been claimed that the reaction is first order with respect to MnO_4^- .

State whether you agree or disagree with this claim. Use evidence from your graph to support your answer.

.....

 [2]

- (b) The uncertainty associated with a burette reading is $\pm 0.05 \text{ cm}^3$. Calculate the percentage uncertainty of your least accurate titre obtained in experiment (a).

[1]

[Total: 13]

3 Qualitative analysis

(a) **FA 9** is a double salt which contains two cations and a single anion from those listed in the Qualitative Analysis Notes on pages 15 and 16.

Carry out the following tests carefully and record your observations in **Table 3.1**.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved.

Table 3.1

test	observations
(i) Place a spatula full of FA 9 into a dry boiling tube. Heat the boiling tube gently and then strongly. Test for any gas evolved.	
Place a small spatula of FA 9 into a new boiling tube. Add about 6 cm depth of water to dissolve the solid. Use portions of this solution, aqueous FA 9, for tests (ii) – (iv).	
(ii) To a 1 cm depth of aqueous FA 9 in a test-tube, add aqueous sodium hydroxide. Allow to stand.	
(iii) To a 1 cm depth of aqueous FA 9 in a test-tube, add dilute nitric acid followed by aqueous barium nitrate.	
(iv) To a 1 cm depth of aqueous FA 9 in a test-tube, add 5 drops of FA 4 and shake the mixture. Add dilute H_2SO_4 to the mixture.	

[6]

(b) Identify the two cations and the single anion in **FA 9** from your observations.

Cations:

Anion:

[2]

(c) Suggest an explanation for your observations in test (ii), in terms of the chemical changes occurring and identity of products formed.

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..... [2]

(d) A student claimed that **FA 9** contains an ion which is a reducing agent.

(i) Describe a test, using only the bench reagents provided which will allow you to verify the claim made by the student.

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..... [1]

(ii) Perform the test you have described in (d)(i). Record and use your observations to explain whether the claim made by the student is correct.

[1]

(e) Investigation of some reactions of FA 10

FA 10 is a neutral-organic compound with 2 functional groups. It has a molecular formula $C_9H_9NO_2$ and it can form intramolecular hydrogen bonding.

The following tests, (i) to (iv) have been carried out on **FA 10** and their observations are recorded in **Table 3.2**.

Table 3.2

test	observations
(i) To 2 cm ³ of 2,4-DNPH, add 3 drops of FA 10 .	An orange precipitate is formed
(ii) To 5 drops of FA10 , add 5 drops of iodine solution. Then add dilute sodium hydroxide until the colour of iodine is discharged. Warm in a water bath.	A pale yellow ppt is formed.
(iii) To a 1 cm depth of aqueous silver nitrate in a test-tube, add a few drops of aqueous sodium hydroxide and then add aqueous ammonia slowly until the grey precipitate that forms just dissolves. To this solution, add 1 cm depth of FA 10 and leave to stand in a warm water bath.	No observable change
(iv) To a 2 cm depth of FA 10 , add an equal volume of HCl(aq). Heat the solution.	Two immiscible layers formed. A homogeneous solution is obtained.

(f) State and explain the chemical change iodine underwent during the reaction in test e(ii).

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[1]

(g) Based on the information given above, deduce a structure for **FA 10**, and give 3 pieces of evidence from the observations in **Table 3.2** to explain your deduction, including the chemistry of the reactions.

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Structure of **FA 10**:

[4]

[Total: 17]

4 Planning

There are four colourless solutions, labelled as **A1**, **A2**, **B1** and **B2**.

A1 and **A2** could be $1.0 \text{ mol dm}^{-3} \text{H}_2\text{SO}_4$ or $2.0 \text{ mol dm}^{-3} \text{H}_2\text{SO}_4$.

B1 and **B2** are either $1.0 \text{ mol dm}^{-3} \text{NaOH}$ or $1.0 \text{ mol dm}^{-3} \text{Ba(OH)}_2$.

- (a) By using only the chemicals above, suggest how Ba(OH)_2 can be identified. Support your answer with the aid of an equation including state symbols. Do not carry out this experiment.

.....

[2]

Heat is evolved when dilute H_2SO_4 and aqueous NaOH are mixed. The maximum temperature change of the mixture depends on the amount of water formed and its total volume. Using appropriate volumes of reagents, the maximum temperature change can be used to identify the two solutions, **A1** and **A2**.

- (b) Write a balanced equation for the complete reaction of aqueous H_2SO_4 and aqueous NaOH .

.....[1]

- (c) Plan an investigation to identify **A1** and **A2** by determining the maximum temperature change, ΔT_{max} when the two acids, **A1** and **A2** are separately neutralised by aqueous sodium hydroxide.

The total volume of the resultant mixture should be constant for all experiments.

You may assume that you are only provided with:

- 250 cm^3 of $1.0 \text{ mol dm}^{-3} \text{NaOH(aq)}$
- 250 cm^3 of $1.0 \text{ mol dm}^{-3} \text{H}_2\text{SO}_4(\text{aq})$
- 250 cm^3 of $2.0 \text{ mol dm}^{-3} \text{H}_2\text{SO}_4(\text{aq})$
- a thermometer
- Styrofoam cup
- common apparatus in the laboratory

Note: No pH indicators are provided.

In your plan, you should include brief details of:

- the apparatus you would use,
- the quantities of reagents you would use,
- the procedure you would follow and the measurements you would make,
- how you would determine the identity of **A1** and **A2**.

Assume that 4.18 J of heat energy raises the temperature of 1.0 cm^3 of the mixture by $1.0 \text{ }^\circ\text{C}$.

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.....[6]

- (d) Show how the enthalpy change of neutralisation, ΔH_{neu} can be calculated from the data of one of the experiments in your plan.

[2]

- (c) Explain whether the enthalpy change of neutralisation will be more exothermic, less exothermic or remain the same in the following situations:

- (i) hydrochloric acid was used instead of sulfuric acid;

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..... [1]

- (ii) ethanedioic acid was used instead of sulfuric acid.

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..... [1]

[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

<i>anion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids)

(c) Tests for gases

<i>gas</i>	<i>test and test result</i>
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

<i>halogen</i>	<i>colour of element</i>	<i>colour in aqueous solution</i>	<i>colour in hexane</i>
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

