



CANDIDATE
NAME

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CHEMISTRY

9729/04

Paper 4 Practical

20 August 2024

2 hours 30 minutes

Candidates answer on the Question Paper.

READ THESE INSTRUCTIONS FIRST

Write your **name**, **CT group**, **centre number** and **index number** 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.

DO **NOT** WRITE ON ANY BARCODES.

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	
2	
3	
4	
Total	

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

1 Qualitative Analysis of FA 1

FA 1 is an aqueous solution containing two cations & one anion.

FA 2 is hydrogen peroxide.

You will perform tests to identify the cations and anion in **FA 1**.

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

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

Where gases are released, they should be identified by a test, **described in the appropriate place in your observations**.

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

No additional tests for ions present should be attempted.

(a) (i) Carry out the following tests. Carefully record your observations in Table 1.1.

Table 1.1

Note: Observations to test 1 have been recorded for you.

test		observations
1	To 1 cm depth of FA 1 in a test-tube, add one spatula full of solid sodium carbonate.	Effervescence of colourless and odourless gas that gives white ppt in limewater. White ppt observed after effervescence ceased.
2	Place 1 cm depth of FA 1 in a boiling tube using a dropping pipette. Measure 6 cm ³ of aqueous sodium hydroxide with a measuring cylinder. Transfer 1 cm ³ of aqueous sodium hydroxide to FA 1 in the boiling tube using a dropping pipette. Mix the contents of the tube thoroughly. Repeat this with another 1 cm ³ of aqueous sodium hydroxide. Repeat until the measuring cylinder is empty.	

	Filter the mixture into a clean test-tube using a filter funnel. While waiting, proceed to test 3.	
	Transfer 1 cm depth of the filtrate to a test-tube and add dilute sulfuric acid (FA 4) dropwise until no further changes are seen.	
	<p>If the earlier filtration is not completed, carefully decant the remaining liquid into the sink. Wash the residue by adding deionised water to the filter funnel until it is around half full.</p> <p>Wait for around one minute before carefully decanting the remaining liquid into the sink. Retain the residue for use in (b).</p>	
3	<p>To 1 cm depth of FA 1 in a test-tube, add aqueous ammonia, dropwise with shaking.</p> <p>Continue adding aqueous ammonia until no further changes are seen.</p> <p>Leave the test-tube to stand.</p>	
4	<p>To 1 cm depth of FA 1 in a test-tube, add 1 cm depth of aqueous $\text{Ba}(\text{NO}_3)_2$.</p> <p>Add dilute HNO_3 to the above.</p>	

[6]

- (ii) Identify the two cations and one anion in **FA 1**. For each ion, complete Table 1.2 by stating evidence from tests 2 to 4.

Table 1.2

cation	evidence

anion	evidence

[3]

- (iii) Based on the given observations in test 1, describe the types of reaction that occurred and identify any new substances formed.

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.....[2]

- (b) (i) Carry out the following tests on the residue from **test 2** in **1(a)**.

test	observations
Place the filter funnel with the washed residue from (a) over a clean test-tube. Carefully add dilute HNO_3 to the filter funnel until it is around half full. Collect around 1 cm depth of the filtrate.	
To the 1 cm depth of the filtrate in the test-tube, add 1 cm depth of FA 2 . Then, add aqueous sodium hydroxide drop by drop carefully , with shaking, until no further changes are seen.	

[3]

- (ii) By considering your observations in **1(b)(i)**, explain how the addition of aqueous sodium hydroxide affects the reaction involving **FA 2**.

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.....[1]

[Total: 15]

2 To study the kinetics of the reaction between potassium manganate(VII) and ethanedioic acid in the presence of FA 1

You are provided with the following.

FA 3 is 0.100 mol dm⁻³ potassium iodide.

FA 4 is 1.00 mol dm⁻³ sulfuric acid.

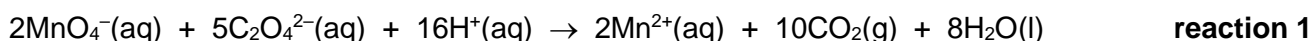
FA 5 is 0.0050 mol dm⁻³ sodium thiosulfate.

FA 6 is 0.0200 mol dm⁻³ potassium manganate(VII).

FA 7 is 0.200 mol dm⁻³ ethanedioic acid.

Solution I is starch indicator.

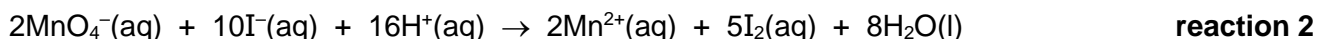
Manganate(VII) ions react with ethanedioate ions in acidic solution as follows.



Part I

You will prepare a reaction mixture containing potassium manganate(VII), ethanedioic acid, sulfuric acid and **FA 1**.

At suitable time intervals, you will withdraw aliquots of the reaction mixture and add them to potassium iodide solution (in excess). The MnO_4^- ions (remaining in each aliquot at that time) react with iodide ions as follows.



You will then titrate the iodine produced against sodium thiosulfate solution, and the reaction is as follows.



You will analyse the results using a graph of volume of sodium thiosulfate against time.

- (a) *You are advised to study this entire series of steps before starting your experiment. You are also advised to manage your time effectively so that you can complete all the titrations and the question parts in the next few pages.*

Measuring and filling of FA 3 and FA 5

- 1 Use a measuring cylinder to add about 10 cm³ of **FA 3** into each of the five labelled boiling tubes.
- 2 Fill a burette with **FA 5**.

Preparation of reaction mixture and withdrawing aliquots

- 3 Use separate measuring cylinders to add the following into the conical flask labelled 'reaction mixture'.
 - 48.0 cm³ of deionised water
 - 50.0 cm³ of **FA 7**
 - 5.0 cm³ of **FA 4**
 - 2.0 cm³ of **FA 1**

- 4 Use another measuring cylinder to measure 20.0 cm^3 of **FA 6**. Add this **FA 6** into the mixture prepared in step 3, immediately starting the stopwatch when the two liquids contact. Swirl the reaction mixture thoroughly.
- 5 At approximately 1 minute, withdraw a 10.0 cm^3 aliquot of the reaction mixture to a 10 cm^3 measuring cylinder, using a dropping pipette. Immediately add this aliquot into boiling tube 1 (containing **FA 3**) and shake the sample carefully to prevent spillage. Record the transfer time in minutes and seconds, to the nearest second, when the aliquot is added.
- 6 At approximately 2 minutes, repeat step 5 but add the aliquot into boiling tube 2.
- 7 Choose three other time intervals at about 2-minute intervals. You should **not** exceed a maximum reaction time of 10 minutes. At each of your chosen times, repeat step 5 but add each aliquot into the corresponding boiling tube.

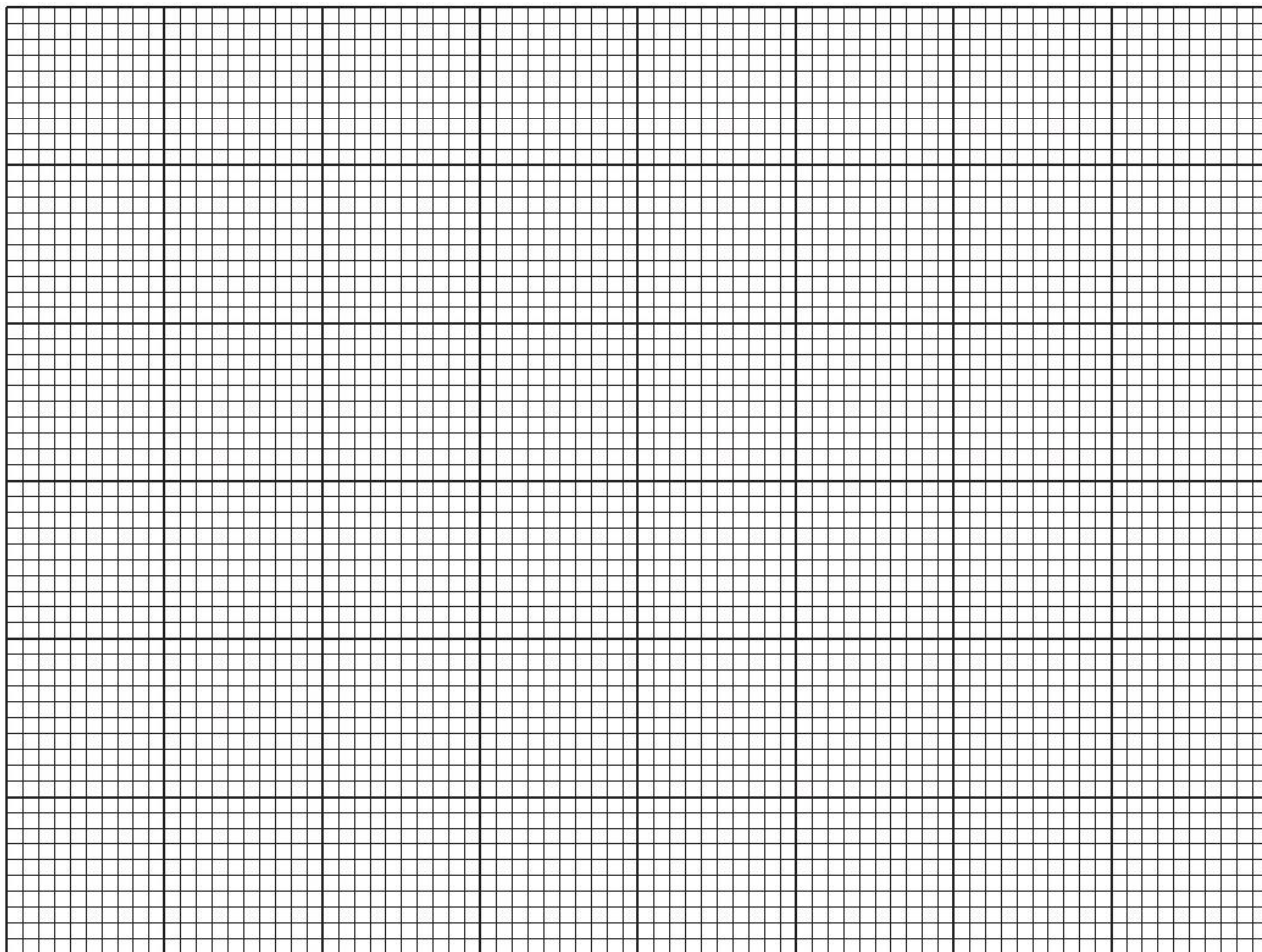
Titration of withdrawn samples

- 8 Add all of the contents of boiling tube 1 into a clean conical flask. Rinse this boiling tube with deionised water, and add the washings into the same conical flask.
- 9 Titrate the iodine in this solution with **FA 5** until the solution is pale yellow. Add 1 cm^3 of **Solution I** into this solution and continue titrating until the point when the solution just turns colourless. The end-point of this titration is expected to be less than 20 cm^3 of **FA 5**. Record your titration results.
- 10 After your titration, wash this conical flask thoroughly with water.
- 11 Repeat steps 8 to 10 for each of the other boiling tubes. *Note that each titration can only be performed once, and you are only given one conical flask for the titrations.*

Record all your experimental results below. In addition, you are to convert the transfer times to decimal values, t , in minutes, recorded to one decimal place. For example, a transfer time of 1 min 28 s becomes $1 \text{ min} + 28/60 \text{ min} = 1.5 \text{ min}$.

[3]

- (b) On the grid below, plot a graph of volume of **FA 5** on the y -axis, against t on the x -axis. Label the axes clearly. Draw a smooth curve taking into account all of the plotted points (but ignoring any anomalous points).



[3]

- (c) It has been claimed that the order of reaction with respect to MnO_4^- is one.

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

.....

[2]

(d) In the following questions, you will use your graph in (b) to determine the rate of change of the concentration of MnO_4^- ions in the reaction mixture at $t = 5$ min.

(i) Draw a tangent to your curve at $t = 5$ min. [1]

(ii) Determine the gradient of your tangent, showing clearly how you obtained your answer.

gradient of tangent = $\text{cm}^3 \text{ min}^{-1}$ [1]

(iii) Calculate the rate of change in amount of $\text{S}_2\text{O}_3^{2-}$ ions needed for complete reaction at this time.

rate of change in amount of $\text{S}_2\text{O}_3^{2-}$ ions = mol min^{-1} [1]

(iv) Calculate the rate of change in amount of MnO_4^- ions remaining in 10 cm^3 of the reaction mixture at this time.

rate of change in amount of MnO_4^- ions in 10 cm^3 = mol min^{-1} [1]

(v) Hence, calculate the rate of change of the concentration of MnO_4^- ions in the reaction mixture at this time.

rate of change of the concentration of MnO_4^- ions = $\text{mol dm}^{-3} \text{ min}^{-1}$ [1]

Part II

- (e) A similar experiment was performed by a student but without adding **FA 1**. Also, a total of 50.0 cm^3 of deionised water was added instead so that the reaction mixture has the same volume as in Part I. The results obtained are shown in Fig. 2.1.

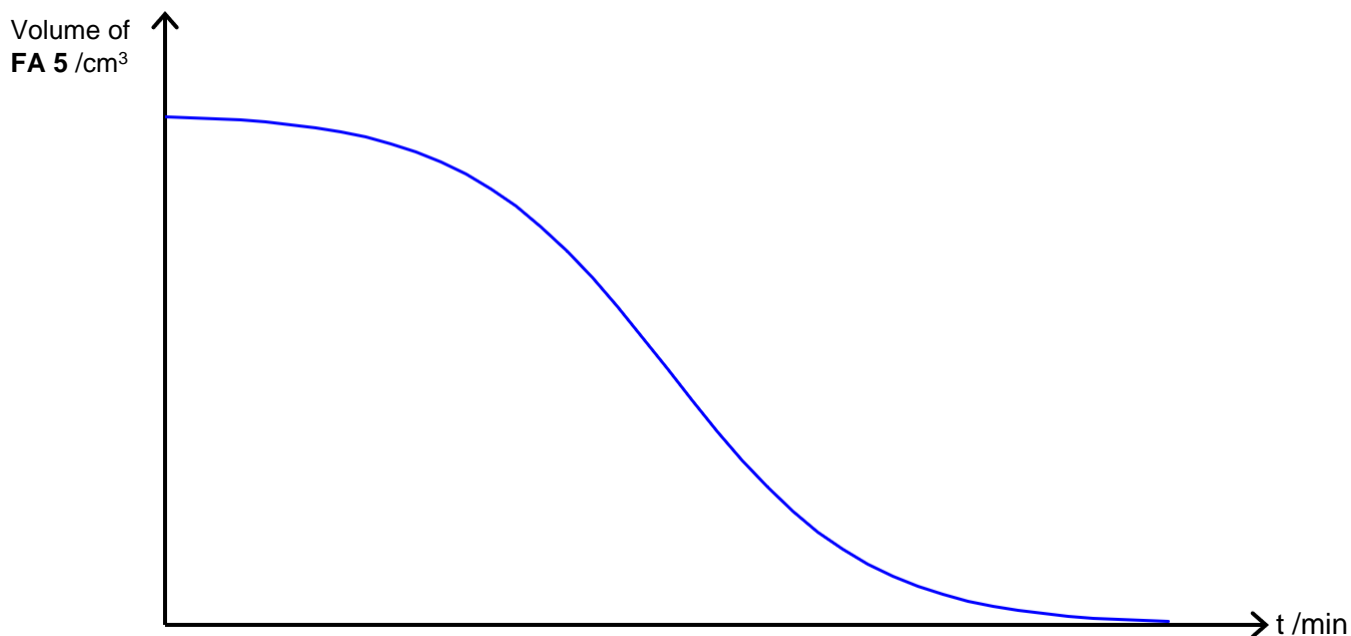


Fig. 2.1

- (i) Describe the shape of this line, linking to how the rate changes during the reaction.

.....

[2]

- (ii) Explain why the graph in Fig. 2.1 has a different shape from the one you plotted in **2(b)**.

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[2]

[Total: 17]

3 Determination of percentage purity by mass of a sodium carbonate, Na_2CO_3 , sample.

FA 4 is 1.00 mol dm^{-3} sulfuric acid.

FA 8 contains a sodium carbonate solution of unknown concentration.

Solution II is methyl orange indicator.

In this question, you will perform a titration to determine the concentration of a sodium carbonate solution.

(a) (i) Titration of **FA 8** against **FA 4**.

1. Fill the burette with **FA 4**.
2. Use the pipette to transfer 25.0 cm^3 of **FA 8** into a 250 cm^3 conical flask.
3. Add a few drops of **Solution II** into the same conical flask.
4. Titrate the mixture in the conical flask until the end-point is reached.
5. Record your titration results, to an appropriate level of precision, in the space provided below.
6. Repeat steps 2 to 5 until consistent results are obtained.

Titration Results

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

[3]

- (ii) From your titrations, obtain a suitable volume of **FA 4** to be used in your calculations. Show clearly how you obtained this value.

volume of **FA 4** = [3]

- (b) (i) Write the balanced equation for the reaction between sodium carbonate and sulfuric acid.

..... [1]

- (ii) **FA 8** was initially prepared by dissolving 90.0 g of an impure sample of sodium carbonate in 1.00 dm³ of water. Calculate the percentage purity by mass of the original sample.
[A_r: H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

[4]

[Total: 11]

4 Planning: Determining the enthalpy change of combustion of ethanol

The combustion of ethanol, $\text{CH}_3\text{CH}_2\text{OH}$, is an exothermic process. The heat evolved during combustion can be used to heat up a fixed amount of water in a copper can. By measuring the temperature rise of the water, the enthalpy change of combustion of ethanol, ΔH_c , can be determined.

However, the heat absorbed by the copper can must be taken into account in order to obtain a more accurate result.

The heat capacity of a copper can, C , is defined as the amount of heat needed to raise the temperature of the copper can by 1 K. Its units is given as J K^{-1} .

The value of C can be determined experimentally by mixing a known volume of hot water into a known volume of cold water in a copper can. The difference between heat lost by the hot water and heat gained by the cold water is the heat absorbed by the copper can.

Use the following information for your calculations:

- specific heat capacity of water = $4.18 \text{ J g}^{-1} \text{ K}^{-1}$
- density of water = 1.00 g cm^{-3}

- (a) A student performed an experiment to determine the heat capacity of the copper can, C . The student poured 75 cm^3 of hot water into the copper can which already contained 75 cm^3 of cold water. The final temperature of the water in the copper can was measured. The results are shown in Table 4.1.

Table 4.1

Temperature of cold water and copper can before mixing / $^{\circ}\text{C}$	8.9
Temperature of hot water before mixing / $^{\circ}\text{C}$	60.4
Final temperature of water and copper can after mixing / $^{\circ}\text{C}$	25.6

Determine the value of C in J K^{-1} .

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..... [3]

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..... [6]

(c) Describe how you would use the results obtained in (a) and (b) to determine ΔH_c .

State an assumption that you have made in your calculations.

[M_r of ethanol = 46.0]

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..... [3]

[Total: 12]

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) Test 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