	Anglo-Chinese Junior C JC2 Preliminary Examination Higher 2	ollege	A Methodist Institution (Founded 1886)
CANDIDATE NAME		FORM CLASS	
TUTORIAL CLASS		INDEX NUMBER	

CHEMISTRY

Paper 4 Practical

9729/04 1 August 2023 2 hours 30 minutes

Candidates answer on the Question Paper.

READ THESE INSTRUCTIONS FIRST

Write your index number and name 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 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.

Shift	
Laboratory	

For Examiner's Use		
1	/ 16	
2	/ 19	
3	/ 20	
Total	/ 55	

This document consists of 20 printed pages and 2 blank pages.

9729/04/Prelim/2023

1 Determination of the change in the oxidation number of a transition metal ion, X²⁺

FA 1 is 0.0125 mol dm⁻³ potassium manganate(VII), KMnO₄ FA 2 is 0.0720 mol dm⁻³ transition metal salt, XSO₄ FA 3 is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄

You are to determine, by titration, the change in the oxidation number of a transition metal ion, X^{2+} , upon oxidation by acidified potassium manganate(VII).

(a) Procedure

- 1. Fill the burette with **FA 1**.
- 2. Using the pipette, transfer 25.0 cm^3 of **FA 2** into the conical flask.
- 3. Using a measuring cylinder, transfer 10.0 cm³ of **FA 3** to the same conical flask.
- 4. Titrate the mixture in the conical flask with **FA 1**. The end-point is reached when the first permanent pale pink colour is seen.
- 5. Record your titration results, to an appropriate level of precision, in the space provided.
- 6. Repeat steps 2 to 5 to obtain consistent results.
 - (i) Titration results

[2]

(ii) From your titration results in (a)(i), obtain a suitable volume of FA 1, V_{FA 1}, to be used in your calculations. Show clearly how you obtained this volume.

V_{FA 1} =[4]

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(b) (i) Calculate the amount of MnO_4^- present in $V_{FA,1}$ obtained in (a)(ii).

amount of MnO_4^- =[1]

(ii) Calculate the amount of X^{2+} in 25.0 cm³ of **FA 2**.

(iii) The half-equation for the reduction of MnO_4^- is shown.

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

It is known that X²⁺ is oxidised to either X³⁺ or X⁴⁺, but not both.

Determine the stoichiometric ratio of:

- MnO₄⁻ to X²⁺ if X³⁺ is formed,
- MnO_4^- to X^{2+} if X^{4+} is formed.

 MnO_4^- : X²⁺ if X³⁺ is formed =

 MnO_4^- : X²⁺ if X⁴⁺ is formed =[1]

(iv) Using your answers from (b)(i) to (iii), determine the change in the oxidation number of X²⁺ upon oxidation by acidified MnO₄⁻.

[Turn over

(v) Hence, write a balanced equation, with state symbols, for the oxidation of X²⁺ by acidified MnO₄⁻.

......[1]

(c) A student claimed that using a burette to measure 25.0 cm³ of **FA 2** in *Step 2* would give a more accurate result than using a pipette.

The maximum error in each volume reading on a burette is ± 0.05 cm³. The percentage error incurred using a 25.0 cm³ pipette is 0.24%.

By calculating the percentage error incurred using a burette, deduce if the claim by the student is correct.

(d) Explain whether using a burette instead of a measuring cylinder to measure 10.0 cm³ of FA 3 in Step 3 would affect the accuracy of the experimental results.

.....[1]

(e) Table 1.1 shows electrode reactions with their standard electrode potential values. **Table 1.1**

electrode reaction	E ^o / V
$X^{3+} + e^- \Rightarrow X^{2+}$	+0.77
$X^{4+} + 2e^- \rightleftharpoons X^{2+}$	+0.86
$MnO_4^- + 8H^+ + 5e^- \rightleftharpoons Mn^{2+} + 4H_2O$	+1.52
$NO_3^- + 3H^+ + 2e^- \rightleftharpoons HNO_2 + H_2O$	+0.94

Use the relevant data to explain, with reference to the expected titre readings, whether nitric acid can be used in place of sulfuric acid in the oxidation of X^{2+} by MnO₄⁻.

[Total: 16]

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2 Determination of the kinetics of the iodination of propanone

The rate of a chemical reaction is affected by the concentration of reactants according to the rate equation which has to be determined experimentally.

In this experiment, propanone is reacted with iodine in the presence of an acid catalyst.

 $CH_3COCH_3 + I_2 \longrightarrow CH_3COCH_2I + HI$

The order of reaction with respect to iodine can be found by titrating the concentration of iodine remaining at regular time intervals with sodium thiosulfate solution.

FA 3 is 1.00 mol dm⁻³ sulfuric acid, H_2SO_4 **FA 4** is 1.00 mol dm⁻³ propanone, CH_3COCH_3 **FA 5** is a solution of iodine dissolved in potassium iodide solution **FA 6** is 0.010 mol dm⁻³ sodium thiosulfate, $Na_2S_2O_3$ **FA 7** is 0.05 mol dm⁻³ sodium hydrogencarbonate, NaHCO₃ starch indicator

- (a) **Procedure**:
 - **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 this experiment.

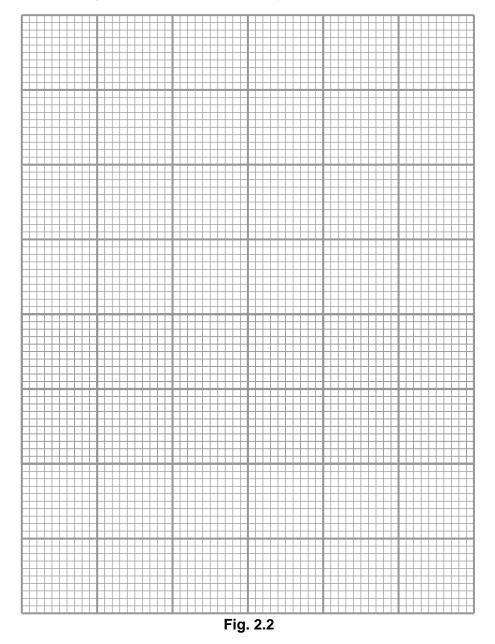
- 1. Fill the burette labelled **FA 6**, with **FA 6**.
- 2. Using a measuring cylinder, add 25.0 cm³ of **FA 3** to the conical flask labelled **reaction mixture**. Using the same measuring cylinder, add 25.0 cm³ of **FA 4** into the same conical flask.
- 3. Using a measuring cylinder, add 50.0 cm³ of **FA 5** to the same conical flask. Insert the stopper, start the stopwatch, and swirl the mixture to mix its contents.
- 4. Using a measuring cylinder, add 10.0 cm³ of **FA 7** to a second conical flask.
- 5. At approximately 5 minutes, pipette 10.0 cm³ of the reaction mixture into the second conical flask and vigorously swirl the mixture. Read and record the actual time of transfer in minutes and seconds, to the nearest second, when the aliquot is completely transferred to the second conical flask.
- 6. **Immediately** titrate the I₂ in the second conical flask with **FA 6** until the solution turns yellow. Using a dropping pipette, add 1 cm³ of starch indicator and continue titrating until the solution becomes colourless. Record your titration results.
- 7. Repeat steps **4** to **6** at approximately 10, 15, 20 and 25 minutes.
- Record your results in Table 2.1 provided. In addition, you should convert all values of actual time to a decimal time, *t*, in minutes, recorded to 1 decimal place. For example, an actual time of 5 min 11 s becomes 5 min + 11/60 min = 5.2 min.

Results

Table 2.1

time / min	actual time / min and s	decimal time, t / min	initial burette reading / cm ³	final burette reading / cm ³	volume of FA 6 used / cm ³
5	1 11				
10	, ,				
15	1 11				
20	1 11				
25	1 11				
					[3]

(b) Plot a graph of volume of **FA 6** used against decimal time, t, on the grid in Fig. 2.2. Your scale on the *y*-axis should allow for extrapolation. Start the *x*-axis at time = 0.





(c) (i) Write an equation for the reaction between iodine and thiosulfate ions.[1] (ii) State the relationship between concentration of iodine remaining in the reaction mixture at regular time intervals and the volume of FA 6 used.[1] (iii) A student stated that the determination of actual time in step 5 is of low accuracy. Suggest a modification to the procedure in steps 4 and 5 to improve the accuracy of actual time determined.[1] (d) Using the graph plotted, answer the following questions about the reaction between iodine and propanone. Deduce the order of reaction with respect to the concentration of iodine. (i)[1] (ii) Explain the significance of the intercept of the graph with the y-axis.[1] (iii) Calculate the initial concentration of iodine in FA 5.

(e) Planning

In determination of the kinetics of the iodination of propanone, we have only deduced the order of reaction with respect to iodine thus far.

The procedure in **2(a)** can be modified to investigate the order with respect to propanone and the acid catalyst, and then obtain the experimentally determined rate equation.

Plan an investigation, based on the experiment described in **2(a)**, to determine the order of reaction with respect to propanone.

You may assume that you are provided with

- the same chemicals and their respective quantities in 2(a),
- a 50 cm³ volumetric flask, and
- the equipment normally found in a school or college laboratory.

In your plan you should include brief details of

- the reactants that you would use,
- the apparatus that you would use in addition to that specified in 2(a),
- the procedure that you would follow and the measurements that you would take,
- how you would determine the order of reaction with respect to propanone.

Note:

For similar procedural steps in 2(a), you may quote "Repeat steps ... to ... in 2(a)."

.....[6] [Total: 19]

3 Organic Analysis

Before starting this analysis, please ensure that your Bunsen burner is turned off.

In this question you will deduce the identities of four organic compounds, **FA 8**, **FA 9**, **FA 10**, and **FA 11**. You will perform a series of test-tube reactions and use your observations to help you distinguish between the compounds.

In addition to having access to the usual bench reagents, you are also provided with the following:

- iodine solution; and
- aqueous bromine located in the fume cupboard.
- (a) You are provided with the liquid samples of **FA 8**, **FA 9** and **FA 11**, each of which, together with **FA 10**, is one of the following compounds.

propanone cyclohexene ethanal 2-methylpropan-2-ol

FA 10 is not provided; observations for **FA 10** have already been recorded in Table 3.1. Use these provided observations for **FA 10** and your own observations to deduce the identities of **FA 8**, **FA 9**, **FA 10** and **FA 11**.

(i) Perform the tests described in Table 3.1. Record your observations in the table. Test and identify any gases evolved.

Use a fresh sample of each liquid in each test. Samples of FA 8, FA 9 and FA 11 are provided in sample tubes. It is essential that you replace the lid as soon as you have removed the sample for each test.

		Table 3.1		
	Observations with FA 8	Observations with FA 9	Observations with FA 10	Observations with FA 11
Add 5 drops of FA 8 into a clean test-tube.				
To this test-tube, add about 1 cm depth of bromine water. Shake the contents vigorously.			Solution remains orange.	
Repeat using FA9 and FA 11 , in place of FA 8 .				
Add about 1 cm depth of sulfuric acid into a clean test-tube. To this test-tube, add 5 drops of FA 8, followed by 5 drops of KMnO ₄ .				
Repeat using FA9 and FA 11 , in place of FA 8 .			Solution remains purple.	

Add about 1 cm depth of deionised water into a clean test-tube. To this test-tube, add 5 drops of FA 8 and 6			
drops of sodium hydroxide. Now add iodine solution, dropwise, till a yellow colour is present permanently.		No yellow precipitate.	
Prepare a hot water bath.			
Warm the mixture for about 2 minutes.			
Repeat using FA9 and FA 11 , in place of FA 8 .			

[4]

(ii) Complete Table 3.2, using the observations in Table 3.1, to identify the compounds FA 8, FA 9, FA 10 and FA 11.

	Table 3.2
	compound
FA 8	
FA 9	
FA 10	
FA 11	

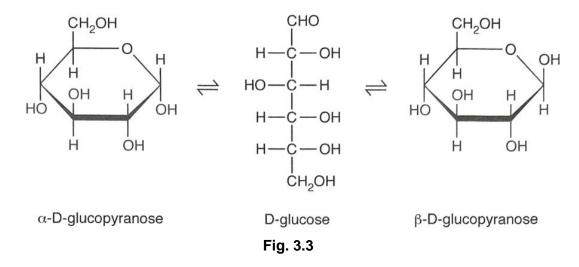
Table	3.2
Iabic	J.Z

[2]

(b) Investigation of some reactions of D-glucose

D-glucose is a sugar with molecular formula $C_6H_{12}O_6$. In aqueous solution, D-glucose exists in a number of isomeric forms.

In aqueous solution, an equilibrium mixture of these isomers is formed. It is thought that at any one time less than 1% of the D-glucose molecules are present in the non-cyclic form, as shown in Fig. 3.3.



FA 12 is 0.5 mol dm⁻³ D-glucose, C₆H₁₂O₆.

(i) Carry out the following test. Carefully record your observations in Table 3.4.

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

	test	observations
1.	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.	
2.	Add aqueous ammonia slowly, with shaking, until the precipitate just dissolves. You may use a clean glass rod to help dissolve the precipitate.	

Table 3	3.4
---------	-----

3.	Add 2 cm depth of FA 12 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.		

- [3]
- (ii) By using two chemical equations, explain the observations in tests 1 and 2.

.....[3] (iii) In (b)(i), the organic product Z can be obtained from D-glucose. Product Z has a non-cyclic structure with molecular formula C₆H₁₂O₇.

Name the type of reaction that D-glucose undergoes in **(b)(i)**. Draw the structure of Z.

(iv) Consider the observations recorded in Table 3.5.

You should not do the following tests. Observations have been recorded for you.

Table 3.5	
test	observations
Add 1 cm depth of FA 12 to a test-tube.	
Add 5 cm depth of 2,4-dinitrophenylhydrazine solution to the test-tube.	no observable change
Observe the mixture for a short time.	
Repeat this test but replace FA 12 with propanone.	yellow precipitate formed

Name the functional group in **FA 12** that can be identified using 2,4-dinitrophenylhydrazine.

.....[1]

(v) Suggest why **FA 12** does not give a positive result with 2,4-dinitrophenylhydrazine, whereas propanone does.

......[1]

Optical activity is the ability of a chiral molecule to rotate the plane of plane-polarised light. It is measured by a polarimeter.

The specific rotation of a chiral molecule is defined as the angle of rotation (in terms of degrees) of the plane of polarisation of a ray of monochromatic light that passes through a 1 decimeter long tube containing the substance at a concentration of 1 gram per dm³. For comparison's sake, the path length is usually fixed.

The specific rotation of α -D-glycopyranose is +112.2°.

The specific rotation of β -D-glycopyranose is +18.7°.

(vi) By examining the signs and the magnitudes of their specific rotations, explain the relationship between the α - and β -D-glycopyranose.

[2]

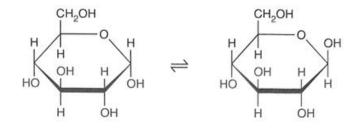
(vii) The sum of the optical activities of each chiral component in a solution is the net optical activity of a solution.

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Consider Fig. 3.3 on page 15 and information on page 18. The interconversion between the three forms of D-glucose is an example of mutarotation, which was discovered in 1846. Mutarotation is a change in the optical rotation of a solution due to a change in the equilibrium between the α and β forms of a carbohydrate, upon dissolution in the aqueous solution.

When a pure sample of either α - or β -D-glycopyranose is dissolved in water, the value of the specific rotation changes over time, eventually reaching the same value of +52.5°.

Calculate the K_c of the following equilibrium.



a-D-glucopyranose

β-D-glucopyranose

 	 [2]

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Qualitative Analysis Notes [ppt. = precipitate]

(a) Reactions of aqueous cations

cation	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, Ba²+(aq)	no ppt. (if reagents are pure)	no ppt.		
calcium, Ca²+(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), Cu²+(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, Mg²⁺(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess		
manganese(II), Mn²+(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, CO3 ²⁻	CO ₂ liberated by dilute acids
chloride, C <i>l</i> ⁻ (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 NH₃(aq))
nitrate, NO₃⁻ (aq)	NH_3 liberated on heating with $OH^-(aq)$ and Al 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 → (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 on warming 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 CO2)		
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