Anglo-Chinese Junior College

JC2 Preliminary Examination
Higher 2

A Methodist Institution
(Founded 1886)

CANDIDATE NAME		FORM CLASS	
TUTORIAL CLASS		INDEX NUMBER	

CHEMISTRY **9729/04**

Paper 4 Practical **5 August 2025**

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 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	**/ 17**
2	**/ 21**
3	**/ 17**
Total	**/ 55**

This document consists of **18** printed pages and **1** blank page.

1 Determination of the stoichiometry of the reaction between hydroxylamine and iron(III) ion

A redox reaction takes place between hydroxylamine, NH_2OH, and iron(III) ion, Fe^{3+}, in an acidic medium. The iron(III) ion is converted to iron(II) ion, Fe^{2+}. The reaction is slow at room temperature, but is completed in a few minutes when heated to 90 °C. The iron(II) ions formed can then be oxidised by manganate(VII) ions for quantitative analysis.

You are to determine, by titration, which of the following **unbalanced** equations represents the reaction between hydroxylamine and iron(III) ion.

equation 1.1 $NH_2OH + Fe^{3+} \rightarrow Fe^{2+} + \ldots N_2 + \ldots H^+ + \ldots H_2O$

equation 1.2 $NH_2OH + 2Fe^{3+} \rightarrow 2Fe^{2+} + \ldots N_2O + \ldots H^+ + \ldots H_2O$

equation 1.3 $NH_2OH + 3Fe^{3+} \rightarrow 3Fe^{2+} + \ldots NO + \ldots H^+$

FA 1 is 0.0150 mol dm^{-3} potassium manganate(VII), $KMnO_4$.

FA 2 is a solution prepared by boiling a 1.00 dm^3 aqueous mixture of 3.30 g of hydroxylamine hydrochloride, $NH_2OH.HCl$, excess iron(III) chloride, $FeCl_3$, and excess sulfuric acid, H_2SO_4.

FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

(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 cm^3 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]

(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 Fe^{2+} in 25.0 cm^3 of **FA 2**.

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

amount of Fe^{2+} = ... [1]

(iii) Calculate the amount of $NH_2OH.HCl$ that reacted in the **FA 2** pipetted into the conical flask.
[A_r: H, 1.0; N, 14.0; O, 16.0; C*l*, 35.5]

Other than the fact that the reaction to prepare **FA 2** was complete and there were no impurities in the chemicals used, state an assumption made in your calculation.

amount of $NH_2OH.HCl$ = ... [2]

assumption ……………………………………………………………………………..

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

(iv) Hence, determine which equation represents the reaction between hydroxylamine and iron(III) ion. Show your working.

equation …………. [1]

(v) From your answer to **(b)(iv)**, write the balanced chemical equation, with state symbols, for the reaction between hydroxylamine and iron(III) ion.

…………………………………………………………………………………….. [2]

(vi) Hence, with reference to the oxidation number of nitrogen, explain whether hydroxylamine acts as an oxidising agent or a reducing agent in the reaction with iron(III) ion.

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………………………………………………………………………………………

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

(d) Another student prepared **FA 2** in a similar way, but found out later that the 3.30 g of hydroxylamine hydrochloride, $NH_2OH.HCl$, contained a small amount of inert impurities.

Suggest what effect this would have on the average titre. Explain your answer.

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…………………………………………………………………………………………….. [2]

[Total: 17]

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2 Determination of a value for the molar enthalpy change of reaction for citric acid and sodium hydrogen carbonate

Citric acid is a weak acid that is found naturally in all citrus fruits.

Citric acid reacts with sodium hydrogen carbonate according to equation 2.1 and produces temperature change.

equation 2.1 $C_6H_8O_7(aq) + 3NaHCO_3(s) \rightarrow Na_3C_6H_5O_7(aq) + 3CO_2(g) + 3H_2O(l)$ $\Delta H_{reaction}$

The maximum temperature change, ΔT_{max}, can be determined by direct measurement of the initial temperature and the final temperature reached.

In this question, you are to plan a procedure that would provide sufficient data to allow you to determine an accurate and reliable value for the molar enthalpy change of reaction, $\Delta H_{reaction}$.

(a) Plan an investigation to determine the maximum temperature change, ΔT_{max}, for the reaction between an aqueous citric acid solution and solid sodium hydrogen carbonate.

Measurements should be taken:
- before the reaction starts,
- during the reaction,
- for some time after the reaction is complete.

You are provided with:
- **FA 4** (approximately 120 cm^3 of 0.100 mol dm^{-3} of citric acid solution)
- **FA 5** (approximately 8 g of solid sodium hydrogen carbonate)
- 250 cm^3 beaker
- 2 polystyrene cups
- 0.2 °C division thermometer
- 50 cm^3 measuring cylinder
- glass rod
- weighing bottle
- weighing balance

In your plan you should include brief details of how you would:
- prepare and set up the apparatus to minimise heat loss,
- measure the initial temperature of the citric acid solution,
- add the sodium hydrogen carbonate **(do not exceed 3 g)** and monitor the temperature change,
- ensure the temperature is recorded accurately,
- repeat the experiment for reliability,
- record and process your data, including how to calculate the average temperature change, $\Delta T_{average}$.

[A_r: C, 12.0; H, 1.0; O, 16.0; Na, 23.0]

[9]

(b) Calculate the average heat change, *q*, for your experiment in **(a)**, and hence determine a value for the enthalpy change of reaction for the reaction between citric acid and sodium hydrogen carbonate reaction, $\Delta H_{reaction,}$ with reference to equation 2.1.

You should assume that:

- specific heat capacity of the mixture is 4.18 J g^{-1} K^{-1};
- density of the mixture is 1.00 g cm^{-3}.

q = ...

$\Delta H_{reaction}$ = ...

[3]

(c) Another student performed a similar experiment that you performed in **(a)**, but measured the temperature at timed intervals. He added 3.00 g of sodium hydrogen carbonate to 50.0 cm^3 of citric acid at t = 30 s and obtained the data shown in Table 2.1 below.

Table 2.1

time/ s	temperature/ °C
0	30.0
10	30.0
20	30.0
30	–
40	29.5
50	28.8
60	27.9
70	27.0
80	26.3
90	25.5
100	24.8
110	24.4
120	24.2
130	24.2
140	24.3
150	24.5
160	24.7

(i) Plot, on Fig 2.1, a graph that the student would obtain using the data in Table 2.1.

Show using construction lines, on Fig 2.1, how the minimum temperature, T_{min}, and the maximum temperature, T_{max}, and ΔT may be obtained at t = 30 s.

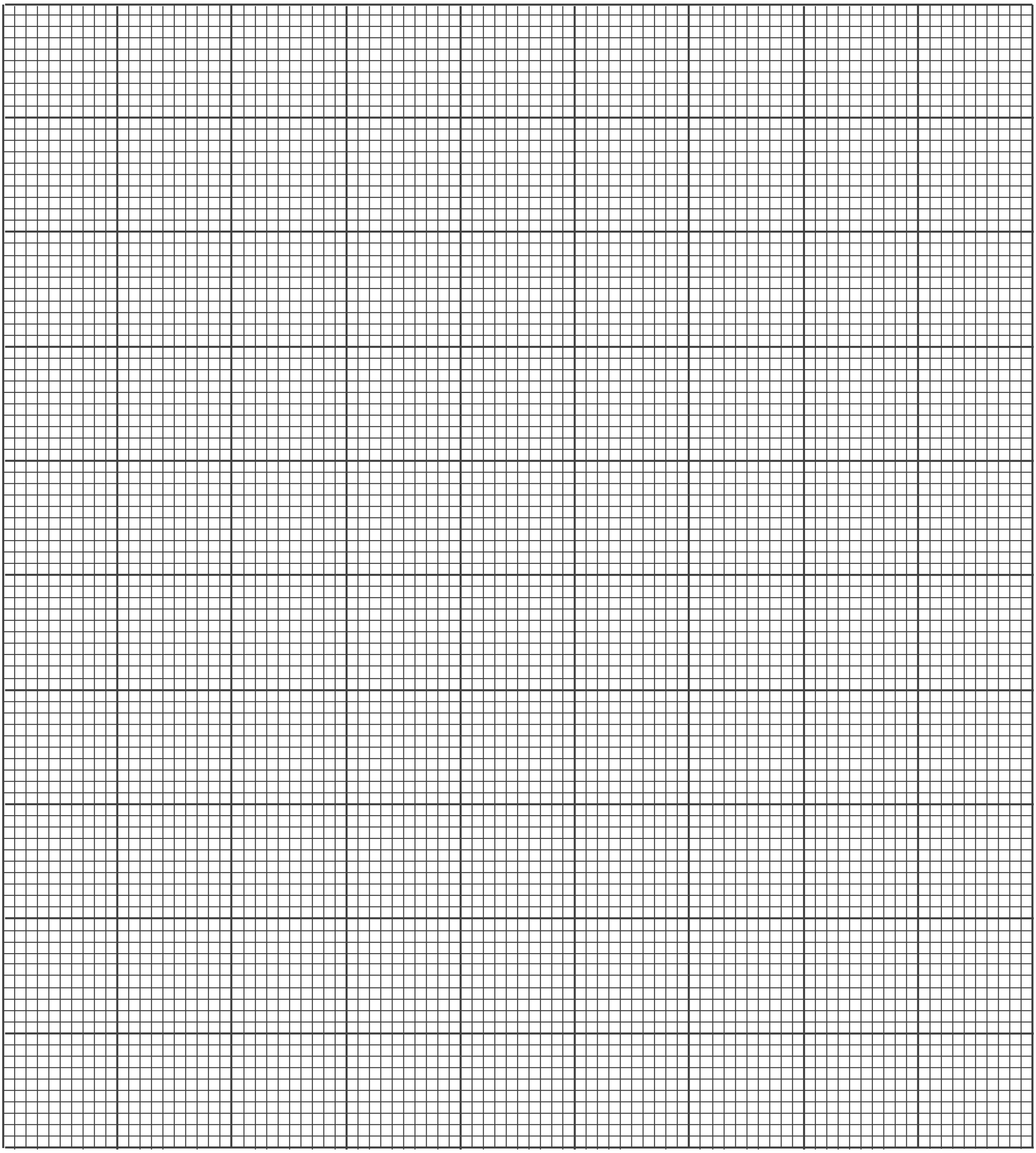

Fig. 2.1

[3]

(ii) From your graph in Fig. 2.1, read the minimum temperature, T_{min} and the maximum temperature, T_{max}, at t = 30 s. Record these values in the spaces provided. Deduce the temperature change at ΔT at t = 30 s.

T_{min} = ...

T_{max} = ...

ΔT = ...

[2]

(iii) Hence, using the graphical method, calculate a value for the enthalpy change of reaction for the reaction between citric acid and sodium hydrogen carbonate reaction, $\Delta H_{reaction}$, with reference to equation 2.1.

$\Delta H_{reaction}$ = ... [2]

(iv) Explain whether the graphical method in **(c)(ii)** gives a more accurate value for ΔT than $\Delta T_{average}$ determined in **(a)**.

....

....

....

....

.... [2]

[Total: 21]

3 Inorganic qualitative analysis

You will carry out tests on **FA 6** and **FA 7**.

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

(a) **(i)** **FA 6** is an aqueous solution containing two cations and one anion.

Carry out the tests in Table 3.1 and record your observations.
Test and identify any gases evolved.

Table 3.1

	tests	observations
1	Test **FA 6** with a blue litmus paper.	
2	To a 0.5 cm depth of **FA 6** in a test-tube, add aqueous sodium carbonate dropwise.	
3	To a 0.5 cm depth of **FA 6** in a test-tube, add aqueous sodium hydroxide dropwise, with shaking, until no further change is seen. Filter the mixture. Retain the filtrate and the residue for use in Tests 4, 5 and 6.	
4	Divide the filtrate from step 3 into two portions. To the **first portion** of the filtrate in a test-tube, add 5 to 6 drops of aqueous NaOH, followed by a spatula full of Zn powder and warm.	
5	To the **second portion** of the filtrate in a test-tube, add aqueous sulfuric acid dropwise, with shaking, until no further change is seen.	
	Add aqueous ammonia to the mixture obtained dropwise, with shaking, until no further change is seen.	

6	Use a spatula to transfer the washed residue from the filter paper to a test-tube. Add aqueous sulfuric acid dropwise, with shaking, until the solid dissolves.	
	Add aqueous potassium iodide dropwise, with shaking, to the solution.	
7	To a 0.5 cm depth of **FA 6**, add aqueous ammonia dropwise until in excess. Filter the mixture. Retain the filtrate for use in Test 8.	
8	To the filtrate in a test-tube, add aqueous sulfuric acid dropwise, with shaking, until no further change is seen.	
	Add aqueous potassium iodide dropwise, with shaking, to the mixture.	

[6]

(ii) From the observations in **(a)(i)**, deduce the identity of the two cations and one anion in **FA 6**.

cations ....

anion .... [2]

(iii) Giving essential supporting evidence from **(a)(i)**, state and explain which tests allow you to deduce the identity of the anion present in **FA 6**.

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…………………………………………………………………………………………….. [2]

(iv) Explain why it would not be meaningful to add Zn powder to a portion of the filtrate obtained in Test 7 to confirm the anion identified in the earlier tests.

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………………………………………………………………………………………………

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

(b) **(i)** **FA 7** is an aqueous solution containing one cation.

Carry out the tests in Table 3.2 and record your observations.

Table 3.2

	tests	observations
1	To a 0.5 cm depth of **FA 7** in a test-tube, add aqueous ammonia dropwise, with shaking, until no further change is seen.	
	Add 1 to 2 spatula full of solid ammonium chloride and shake the mixture.	
2	To a 0.5 cm depth of **FA 7** in a test-tube, add 1 to 2 spatula full of solid ammonium chloride, with shaking, until no further change is seen.	
	Add aqueous ammonia dropwise, with shaking, until no further change is seen.	

[3]

(ii) Explain your observations for Test 1 in **(b)(i)** using the principles of solubility equilibrium.

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……………………………………………………………………………………………….. [2]

(iii) Explain your observations for Test 2 in **(b)(i)** using the principles of solubility equilibrium.

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

[Total: 17]

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 A*l* foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and A*l* 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 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