H

NANYANG JUNIOR COLLEGE
JC 2 PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME	

CLASS		TUTOR'S NAME	

CHEMISTRY **9729/04**

Paper 4 Practical **19 August 2019**

2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name and class 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, 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 18 and 19. 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	**/ 28**
2	**/ 11**
3	**/ 16**
Total	**/ 55**

1 Determination of the percentage by mass of ethanedioic acid

FA 1 is a solution made by dissolving an unknown mass of a mixture of ethanedioic acid, $H_2C_2O_4$, and sodium ethanedioate, $Na_2C_2O_4$.

In aqueous solution, both ethanedioic acid and sodium ethanedioate release all their ethanedioate ions, $C_2O_4^{2-}$. These ions react with manganate(VII) ions as shown.

$$2MnO_4^-(aq) + 16H^+(aq) + 5C_2O_4^{2-}(aq) \rightarrow 10CO_2(g) + 2Mn^{2+}(aq) + 8H_2O(l)$$

You will carry out titrations to find the percentage by mass of ethanedioic acid in the mixture.

FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

(a) (i) Titration of FA 1 against FA 2

1. Fill a burette with **FA 2**.
2. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
3. Use the measuring cylinder to add 30.0 cm^3 of **FA 3** to the same conical flask.
4. Place the conical flask on the tripod and gauze and heat until the temperature of the solution is approximately 70 °C.
5. **Carefully** remove the flask from the tripod.
   Add a few drops of **FA 2** from the burette and swirl the mixture until it turns colourless.

Note: If the reaction mixture does not decolourise or turns brown, reheat it to about 70 °C. Swirl the mixture till it decolourises.
If the brown colour remains, discard the contents of the flask and begin a new titration.

6. Continue the titration until a permanent pale pink is obtained.
7. Record your titration results, to an appropriate level of precision, in the space provided on page **3**.
8. Repeat points 2 to 7 until consistent results are obtained.

Titration results

[2]

(ii) From your titration results, obtain a suitable volume of **FA 2**, $V_{\textbf{FA 2}}$, to be used in your calculations. Show clearly how you obtained this volume.

$V_{\textbf{FA 2}}$ = ... cm^3

[3]

(iii) Calculate the total number of moles of ethanedioate ions present in 25.0 cm^3 of **FA 1**.

moles of ethandioate ions present = ...

[1]

(b) Titration of FA 1 against FA 4

A separate experiment was carried out by titrating **FA 1** against **FA 4** containing 0.0400 mol dm^{-3} sodium hydroxide from a burette.
It was found that 25.0 cm^3 of **FA 1** required **26.75 cm^3** of **FA 4** for complete reaction.

Calculate the number of moles of ethanedioic acid present in 25.0 cm^3 of **FA 1**.

moles of ethandioic acid present = ...

[1]

(c) **(i)** Calculate the mass of sodium ethanedioate, $Na_2C_2O_4$, present in 25.0 cm^3 of **FA 1**.
[A_r: C, 12.0; O, 16.0; Na, 23.0]

mass of sodium ethanedioate present = ...
[1]

(ii) Calculate the mass of ethanedioic acid, $H_2C_2O_4$, present in 25.0 cm^3 of **FA 1**. Hence calculate the percentage by mass of ethanedioic acid in the solid mixture used to prepare **FA 1**.
[A_r: C, 12.0; O, 16.0; H, 1.0]

percentage by mass of $H_2C_2O_4$ = ...
[2]

(d) Explain why the solution in the conical flask was heated to approximatly 70 ^oC before titration.

....

....[1]

(e) Student **B** suggested improving the accuracy of the titration results by decreasing the concentration of **FA 2** to 0.0150 mol dm^{-3}.

State and explain if the student's suggestion would make the titration results more accurate.

....

....[1]

(f) The **FA 2** that was initially added into the conical flask took some time to decolourise. As the titration continued, the rate of decolourisation changed due to the products formed.

To investigate the kinetics of the reaction in part **(a)**, a separate experiment was carried out to observe the rate of carbon dioxide produced over a period of time.

30.0 cm^3 of **FA 2** was added from a measuring cylinder to a conical flask containing 20.0 cm^3 of **FA 1** and 50.0 cm^3 of **FA 3** and the stop watch was started. The volume of carbon dioxide was collected by downward displacement of water using a measuring cylinder and the volume of gas was recorded at 30 s intervals.

(i) Sketch the graph, in **Fig 1.1**, you would expect to obtain for this experiment and use it to explain why **FA 2** decolourises at different rates throughout the titration.

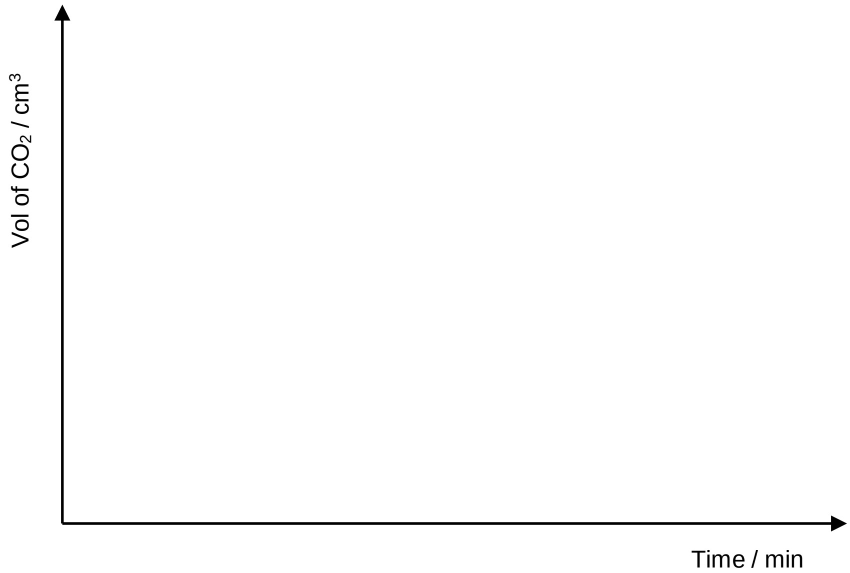

Fig 1.1

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....[3]

(ii) A similar experiment described in **(f)(i)** was carried out using 20.0 cm^3 of **FA 1** with 30.0 cm^3 of another oxidising agent, aqueous potassium persulfate, $K_2S_2O_8$, in the presence of Fe^{3+}(aq) catalyst. The volume of carbon dioxide produced was similarly recorded at 30 s intervals. Sketch in **Fig 1.2,** a graph you would expect for this new experiment

Fig 1.2

[1]

(g) Planning

The kinetics of a chemical reaction can be studied by using the initial rates method. In this method, we note the time taken for a reaction to reach an identification point early in the reaction.

Reaction between iodide ions, I^-, and persulfate ions, $S_2O_8^{2-}$ is an example where its initial rate can be determined.

$$2I^-(aq) + S_2O_8^{2-}(aq) \rightarrow I_2(aq) + 2SO_4^{2-}(aq)$$

If a small and same amount of aqueous sodium thiosulfate is added to every reaction mixture together with starch before the start of the reaction, the iodine produced during the reaction will react immediately with the thiosulfate ions present.

When the small amount of thiosulfate ions have reacted with the iodine produced, a blue-black iodine-starch complex will appear, indicating the experiment has reached the identification point.

By keeping the amount of thiosulfate ions small, the time taken for the reaction to reach the identification point is therefore used to determine the initial rate of that reaction.
The same procedure is repeated with different volumes of reactants and deionised water while keeping the total volume of the reaction mixture constant.

In this question, you are to investigate how the rate of reaction between potassium persulfate and potassium iodide depends on the concentration of potassium persulfate.

(i) You are to plan a series of experiments to determine the time taken for the reaction between potassium persulfate and potassium iodide to reach a same identification point for all the experiments and show how changing the concentration of potassium persulfate will affect the rate of reaction.

You may assume you are provided with:

- 0.60 mol dm^{-3} aqueous potassium iodide, KI,
- 0.20 mol dm^{-3} aqueous potassium persulfate, $K_2S_2O_8$,
- 0.020 ml dm^{-3} aqueous sodium thiosulfate, $Na_2S_2O_3$,
- starch indicator,
- equipment normally found in a school or college laboratory.

A first experiment is carried out using the following quantities
(precision is according to the apparatus used):

25 cm^3 potassium iodide,
40 cm^3 potassium persulfate,
10 cm^3 sodium thiosulfate,
0 cm^3 deionised water,
5 cm^3 starch indicator.

In your plan you should include brief details of:

- the apparatus that you would use,
- the procedure that you would follow,
- how you would ensure your results obtained are reliable,
- a table showing all volumes and precision of each solution you would use and the measurements that you would take.

Table

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....[8]

(ii) Write an ionic equation, including state symbols, for the reaction between the iodine produced and the thiosulfate ions, $S_2O_3^{2-}$, present in the reaction mixture.

....[1]

(iii) Hence calculate the rate of producing iodine if the time taken for experiment 1 was 134 seconds.

[2]

(iv) A preliminary experiment carried out by another student, using approximate volumes of solution, showed that the time taken for an experiment to reach identification point doubled when the potassium persulfate was diluted with an equal volume of water.

Sketch with clearly labelled axes, on **Fig 1.3**, an appropriate graph to represent the results of the student's preliminary experiment.

Fig 1.3

[1]

[Total: 28]

2 Determination of the concentration of sodium hydroxide

When hydrochloric acid is mixed with aqueous sodium hydroxide, the neutralisation reaction releases heat causing a rise in the temperature of the solution.

$$HCl(aq) + NaOH(aq) \rightarrow NaCl(aq) + H_2O(l)$$

In this experiment you will mix different volumes of hydrochloric acid and sodium hydroxide but the total volume will be kept constant. For each mixture you will record the temperature rise.

Since the total volume remains the same, the temperature rise is a direct measure of the heat given out by the reaction. The maximum heat given out occurs when all the acid present is exactly neutralised by all the alkali present. By determining the volumes when this occurs, you can determine the concentration of the sodium hydroxide.

FA 5 is 2.00 mol dm^{-3} aqueous hydrochloric acid, HCl.
FA 6 is aqueous sodium hydroxide, NaOH.

(a) Prepare a table in the space provided on page **12** in which to record, to an appropriate level of precision:

- the volume of **FA 5**, $V_{FA\,5}$,
- the volume of **FA 6**, $V_{FA\,6}$,
- the initial temperature of **FA 5**, T_i,
- the maximum temperature of the solution, T_{max},
- the temperature rise, ΔT, where $\Delta T = T_{max} - T_i$.

Experiment 1

1. Using a measuring cylinder, transfer 40.0 cm^3 of **FA 5** into a polystyrene cup placed in a 250 cm^3 beaker.
2. Record the initial temperature of **FA 5**, T_i, in the space below.
3. Using another measuring cylinder, transfer 10.0 cm^3 of **FA 6** into the same cup.
4. Stir the mixture thoroughly and record in your table the maximum temperature, T_{max} of the solution.
5. Empty the polystyrene cup, rinse thoroughly with water and shake dry.

Experiments 2 to 6

Repeat experiment 1 five times, using 34.0, 28.0, 22.0, 16.0, 10.0 cm^3 respectively, of **FA 5**, at point 1.

In each case, you will need to add sufficient **FA 6** to make sure that the total volume is 50.0 cm^3.

Record all required volumes, temperature taken and calculated values in your table.

Results

[3]

(b) **(i)** Plot a graph of temperature rise, ΔT, on the y-axis against the volume of **FA 5** on the x-axis on page **13**. Draw two best-fit lines taking into account all of your plotted points.

(ii) From your graph, determine the volume of **FA 5** required to exactly react with **FA 6**.

volume of **FA 5** = ...

[4]

(c) **(i)** Calculate the number of moles of hydrochloric acid present in the volume of **FA 5** in **(b)(ii)**.

moles of hydrochloric acid in **FA 5** = ...

[1]

(ii) Calculate the concentration of **FA 6**.

concentration of **FA 6** = ...

[1]

(d) A student decided to modify the original experiment. The total volume of the solution was increased to 70 cm^3 and temperature rises were recorded for 15, 20, 25, 30, 40, 45, 50, 55, 60 and 65 cm^3 of **FA 6**.

Discuss how these two changes would affect the accuracy with which the concentration of **FA 6** could be determined.

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....[2]

[Total:11]

3 Investigation of some organic and inorganic reactions

Carry out the following tests. Carefully record your observations in Tables **3.1** and **3.2**

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

FA 7, **FA 8** and **FA 9** are all solutions of carbohydrates.

- Sugars and starch are carbohydrates.
- Some sugars contain an aldehyde group and hence act as reducing agents.
- Other sugars do not contain an aldehyde group.

Sandell's solution reacts in a similar way to Fehling's reagent.

You will need to heat Sandell's solution in a hot water bath when using it in tests.

Table 3.1

		test	observations		
			FA 7	**FA 8**	**FA 9**
(a)	**(i)**	Add 2 or 3 drops of aqueous iodine.			
	(ii)	Add 2 or 3 drops of acidified potassium manganate(VII) and **allow to stand for two minutes.**			
	(iii)	Add 3 cm depth of Sandell's solution and place the tube in the hot water bath for two minutes.			

[3]

(iv) Identify the following and explain your reasoning:

the carbohydrate that could be starch: ....

explanation :..

....

the carbohydrate that contains an aldehyde group: ....

explanation: ....

....
[2]

(v) Describe a different test, other than using Sandell's solution and Fehling's reagent, that could be carried out to identify the presence of an aldehyde group. Describe how the test would be carried out and the expected observation if the result is positive.

Do not carry out your test.

test: ....

....

observation: ....

....
[1]

FA 10 and **FA 11** are two of the components of Sandell's solution. Each contains one cation and one anion. Carry out the following tests and record all your observations in the table.

Table 3.2

		test	observations FA 10	observations FA 11
(b)	**(i)**	Add a few drops of aqueous silver nitrate.		
	(ii)	Add a few drops of aqueous barium nitrate or aqueous barium chloride, then		
		add dilute nitric acid.		
	(iii)	Add a few drops of aqueous iodine.		
	(iv)	Add a 1 cm depth of aqueous iron(II) sulfate.		
	(v)	Add a 1 cm depth of **FA 11**.		

[6]

(vi) Identify the ions in **FA 10** and **FA 11**.

FA 10 cation: .... anion: ....

FA 11 anion: ....

[2]

(vii) Write an ionic equation, including state symbols, for the reactions of **FA 10** in **(b)(iii)** and **(b)(v)**.

equation for **(b)(iii)**: ....

equation for **(b)(v)**: ....

[2]

[Total:16]

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 $\rightarrow$ (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)

9(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

9(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