HWA CHONG INSTITUTION
C2 Preliminary Examination
Higher 2

NAME		**CT GROUP**	**19S**

CHEMISTRY

9729/04

Paper 4 Practical

25 August 2020

2 hours 30 minutes

Candidates answer on the Question Paper

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 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 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	
2	
3	
4	
Total	

1 Determination of the percentage by mass of ethanedioic acid in a solid mixture

FA 1 is a solution made by dissolving a solid mixture of ethanedioic acid, $H_2C_2O_4$, and sodium ethanedioate, $Na_2C_2O_4$, in water. The percentage by mass of ethanedioic acid in the mixture can be determined by a combination of an acid-base titration (**Titration A**) and a redox titration (**Titration B**). You will carry out **Titration A** only.

FA 2 is 0.0400 mol dm^{-3} sodium hydroxide.
You are also provided with thymol blue indicator.

Titration A

Ethanedioic acid reacts with sodium hydroxide as follows:

$$H_2C_2O_4(aq) + 2NaOH(aq) \rightarrow Na_2C_2O_4(aq) + 2H_2O(l)$$

(a) 1. Fill a burette with **FA 2**.
2. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
3. Add about 10 drops of thymol blue indicator.
4. Add **FA 2** from the burette until the end-point has been reached.
5. Carry out as many titrations as you think necessary to obtain consistent results.

Record your titration results in a suitable format in the space below.

[2]

(b) From your titration results, obtain a suitable value for the volume of **FA 2** needed to react with the ethanedioic acid in 25.0 cm^3 of **FA 1**.

volume of **FA 2** needed = …… cm^3 [3]

(c) **(i)** Calculate the number of moles of sodium hydroxide present in the volume of **FA 2** found in **(b)**.

number of moles of sodium hydroxide = …… mol [1]

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

number of moles of ethanedioic acid = …… mol [1]

Titration B

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 in acidic medium.

(d) Balance the equation for the reaction between ethanedioate ions and manganate(VII) ions in acidic medium.

….. $MnO_4^-(aq)$ + ….. $H^+(aq)$ + 5 $C_2O_4^{2-}(aq)$ → ….. $Mn^{2+}(aq)$ + ….. $CO_2(g)$ + ….. $H_2O(l)$ [1]

25.0 cm^3 of **FA 1** was pipetted into a conical flask. Using a measuring cylinder, 30.0 cm^3 of 1.00 mol dm^{-3} sulfuric acid (an excess) was added. After warming to 70 °C, the aliquot was titrated with potassium manganate(VII). 24.80 cm^3 of 0.0200 mol dm^{-3} $KMnO_4$ was required for complete reaction.

(e) **(i)** Calculate the number of moles of manganate(VII) ions required for complete reaction.

number of moles of manganate(VII) ions = …… mol [1]

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

total number of moles of ethanedioate ions = …… mol [1]

(f) Use your answers to **(c)(ii)** and **(e)(ii)** to calculate the number of moles of sodium ethanedioate, $Na_2C_2O_4$, present in 25.0 cm^3 of **FA 1**.

number of moles of sodium ethanedioate = …… mol [1]

(g) Use your answers to **(c)(ii)** and **(f)** to calculate the percentage by mass of ethanedioic acid in the solid mixture used to prepare **FA 1**. [A_r of H: 1.0; C: 12.0; O: 16.0; Na: 23.0]

percentage by mass of ethanedioic acid = …… % [2]

[Total: 13]

2 Determination of the identities of three salts

You are provided with solutions **FA 3** and **FA 4**, and solid **X** in a capped bottle.

(a) Preparation of FA 5 solution

1. Weigh the capped bottle containing solid **X**.

2. Transfer all the solid into a 250 cm^3 beaker. Add about 100 cm^3 of deionised water. Using a glass rod, stir the solution in the beaker until all the solid dissolves.

3. Transfer the solution to the volumetric flask. Rinse the beaker with deionised water several times, adding each rinsing to the flask.

4. Make up the contents of the flask to the 250 cm^3 mark with deionised water. Stopper and mix thoroughly by inverting the flask a number of times. This solution is **FA 5**.

5. Reweigh the empty bottle and its cap.

In an appropriate format in the space below, prepare a table to record all weighings to an appropriate level of precision.

Note: You will use **FA 5** in **Question 2 and Question 3**.

[1]

FA 3, **FA 4** and the **FA 5** you prepared are salt solutions.
Each contains **one** cation and **one** anion, all of which are **different**.

- **FA 3** or **FA 4** contains a halide ion.
- **FA 5** contains Na^+ cation and a sulfur-containing anion.
- The other five ions are listed in the Qualitative Analysis Notes on pages 18-19.

(b) You will identify the **cations** present in **FA 3** and **FA 4**.

To do this, you will carry out **four** separate tests. You will use aqueous sodium hydroxide, NaOH, and aqueous ammonia, NH_3, separately with **FA 3** and **FA 4**.
Use about 1 cm depth of each salt solution in a test-tube for each test.

If no precipitate forms with aqueous NaOH, transfer the mixture into a **boiling tube** and **carefully** warm the boiling tube and its contents. Do **not** heat until the mixture boils.

Note: Keep the **product mixture P**, obtained from **FA 4** and aqueous NH_3, for use in part **(f)**.

Record **all** of your observations in a table in the space below.

[3]

(c) From the bench reagents provided, choose the reagents you would use to identify the halide ion present in **FA 3** or **FA 4**.

Test **FA 3** and **FA 4** with these reagents and record your observations.

reagents used ……

unknown	observations
FA 3	
FA 4	

[2]

(d) Place about 1 cm depth of dilute sulfuric acid, H_2SO_4, in a test-tube. Add 1 drop of potassium manganate(VII), $KMnO_4$.

Add **FA 5** dropwise until no further change is seen. Record your observation.

unknown	observation
FA 5	

[1]

(e) Use the information given and your observations in **(b)**, **(c)** and **(d)** to identify **four** of the ions. Explain your reasoning, giving the identities of any precipitates formed.

FA 3 contains ……

Explanation ……

……..

……..

FA 4 contains ……

Explanation ……

……..

……..

FA 5 contains ……

Explanation ……

……..

…….. [4]

(f) Place about 1 cm depth of **product mixture P**, obtained in **2(b)**, in a test-tube.
Add solid ammonium chloride, NH_4Cl, a little at a time, until no further change is seen. Stir the mixture with a glass rod after each addition.

Record your observation. Suggest an explanation for the observation, writing equations for the reactions.

Observation ……

Explanation ……

……..

……..

……..

…….. [2]

(g) Planning

The remaining ion that has not been identified contains either carbon or nitrogen.

Use the Qualitative Analysis Notes on pages 18-19 to deduce **three** possible identities of this ion.

possible identities of the ion ……

Describe **two different** tests which would allow you to identify this ion.

In each case, state
- the reagents you would use,
- how you would decide if the test result is positive.

You are not required to carry out your plan.

test 1 ……

……..

……..

……..

test 2 ……

……..

……..

……..…….. [3]

[Total: 16]

BLANK PAGE

3 Investigation of the effect of concentration changes on the rate of a reaction.

Note: You should complete **Question 2(a)** before starting **Question 3**.

Iodate(V) ions, IO_3^-, react with the sulfur-containing ions in **FA 5**, in the presence of acid:

$$IO_3^-(aq) + \text{sulfur-containing ion in } \textbf{FA 5} + H^+(aq) \rightarrow I_2(aq) + \text{other products}$$

The rate of this reaction is affected by the concentration of acid present in the reaction mixture.

In the presence of starch, the iodine produced will give a dark blue colour. Therefore, the effect of the concentration of the acid on the initial rate of the reaction can be studied by measuring the time taken for a fixed amount of iodine to be produced.

FA 5 is the solution from **Question 2(a)** and contains the sulfur-containing ion.

You are also provided with:
FA 6 0.100 mol dm^{-3} dilute sulfuric acid, H_2SO_4
FA 7 3.50 g dm^{-3} aqueous potassium iodate(V), KIO_3
Starch indicator

(a) You are required to perform a series of five 'iodine clock' experiments, by varying the concentration of acid and measuring the time, t, taken for the dark blue colour to appear.

- For each experiment, **solution 1** will contain the same volume of **FA 5** but a different volume of **FA 6**. You will need to ensure that the total volume of **solution 1** is kept constant at 100 cm^3 by adding deionised water as required.

- For each experiment, prepare **solution 2** as described in **(a)(i)**.

Note: Use separate measuring apparatus to prepare **solutions 1** and **2**.

For each experiment, you will note the volume of **FA 6**, $V_{\textbf{FA 6}}$, and the time taken, t, for the reaction mixture to turn dark blue.

You will then calculate the following values to 3 significant figures:
- 1/t,
- lg(1/t),
- lg($V_{\textbf{FA 6}}$).

In the space provided on page 12, prepare a table in which to record, to an appropriate level of precision:
- all volumes used to prepare **solution 1**,
- all values of t,
- all calculated values of 1/t, lg(1/t) and lg($V_{\textbf{FA 6}}$).

(i) Experiment 1

Solution 1

- Pour 15.0 cm^3 of **FA 5** to a 100 cm^3 measuring cylinder and make up the volume to 100.0 cm^3 by adding **FA 6**.

Solution 2

- Pour 15.0 cm^3 of **FA 7** to another 100 cm^3 measuring cylinder and make up the volume to 100.0 cm^3 by adding deionised water.

1. Transfer **solution 1** into a 250 cm^3 beaker.

2. Using a 10 cm^3 measuring cylinder, add 5.0 cm^3 of the starch indicator to the beaker.

3. Pour **solution 2** from the measuring cylinder **rapidly but carefully** into the beaker containing **solution 1**. Start the stopwatch immediately.

4. Stir the mixture using a glass rod.

5. Stop the stopwatch when the dark blue colour first appears.

6. Record the time taken, t, to the nearest second in your table.

7. Wash the beaker thoroughly with water and dry it.

(ii) Experiment 2

8. Prepare a different **solution 1** using 15.0 cm^3 of **FA 5** and 25.0 cm^3 of **FA 6**. Make up the volume to 100.0 cm^3 by adding deionised water.

9. Prepare **solution 2** as described in **experiment 1** above.

10. Based on the procedures described in **experiment 1**, determine the time taken for the solution to turn dark blue, and record it in your table.

11. Wash the beaker thoroughly with water and dry it.

Experiment 1 gives the time taken for the 'fastest' reaction. **Experiment 2** gives the time taken for the 'slowest' reaction.

(iii) Experiments 3 to 5

Choose **three** other suitable volumes of **FA 6**, between 25.0 cm^3 and 85.0 cm^3, for use in the remaining three experiments.

In each case,

- Use 15.0 cm^3 of **FA 5**, together with your selected volume of **FA 6** and deionised water to prepare 100.0 cm^3 of **solution 1**.
- Prepare **solution 2** as described in **experiment 1**.
- Determine the time taken for the solution to turn dark blue.

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

Results

[5]

Graphical determination of order of reaction

In a series of experiments, where the same end-point (i.e. appearance of dark blue colour) is timed, while changing the concentration of one of the reactants and keeping the total volume of the mixture constant,

- 1/t can provide a measure of its initial rate,
- volume of the reactant that is changed in each experiment, can be used as a measure of its initial concentration.

Since the total volume of the reaction mixture is kept constant and only the concentration of **FA 6** has been changed, the rate equation, where ***m*** is the order of reaction with respect to H^+, can be simplified to

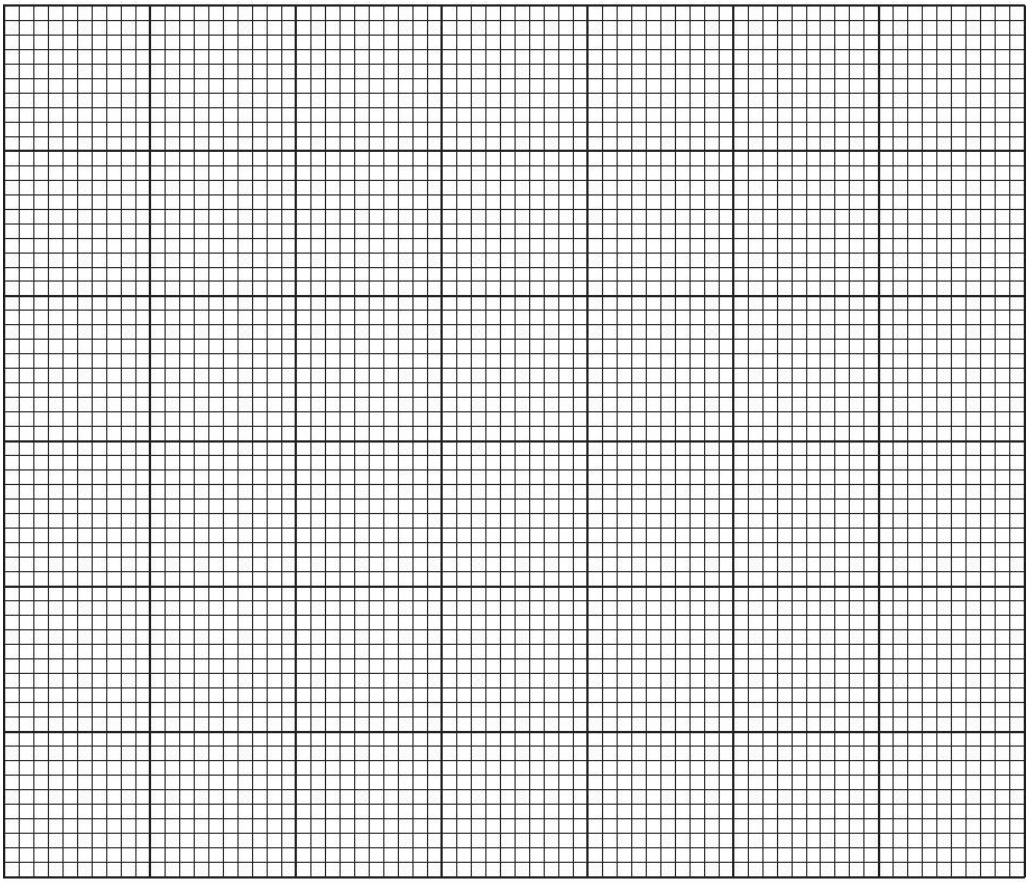
$$\text{rate} = k[H^+]^{m}$$

By taking logarithms of the factors in this equation and by substituting 1/t for rate and $V_{\mathbf{FA\ 6}}$ for concentration respectively,

$$\lg(1/t) = \boldsymbol{m} \times \lg(V_{\mathbf{FA\ 6}}) + \text{constant}$$

Therefore, by plotting a graph of lg(1/t) against $\lg(V_{\mathbf{FA\ 6}})$, you will be able to draw a straight line of best-fit graph, where ***m*** is the gradient of the line.

(b) **(i)** Plot a graph of lg(1/t) on the *y*-axis against lg($V_{\mathbf{FA\ 6}}$) on the *x*-axis. Draw the best-fit straight line taking into account all of your plotted points.



[3]

(ii) Calculate the gradient of the line, and hence, determine the value of ***m***, showing clearly all your working. Give your answer to 3 significant figures.

m = ... [1]

(c) This experiment is known to be very reliable. When it is performed carefully, it is possible to make a reaction mixture that changes colour after a specified time interval.

Use your graph to determine the volume of **FA 6** which would need to be added in **solution 1** so that the reaction mixture turns dark blue after **25 seconds**.

Show clearly your working.

volume of **FA 6** required = ... [2]

(d) Suggest which experiment, from **experiments 1** to **5**, is likely to have the greatest error.

experiment number ……

explanation ……

……

…… [1]

(e) Calculate the concentration of hydrogen ions, $[H^+]$, in mol dm^{-3}, that is present in the reaction mixture for **experiment 1** at time t = 0 s. You may assume that the solutions are perfectly mixed and that the reaction has **not** started at t = 0 s.

$[H^+]$ = …… mol dm^{-3} [2]

(f) Suppose that the following mistake was made in the preparation of **solution 1** for **experiment 2**:

A student added 15.0 cm^3 of **FA 5** to the 100.0 cm^3 measuring cylinder and added **FA 6** up to the 45.0 cm^3 mark on the measuring cylinder. After that, the student made up the volume to 100.0 cm^3 by adding deionised water.

(i) State and explain how the value of t is affected for **experiment 2** described above.

……

……

…… [1]

(ii) Suggest a modification to the experimental procedure to avoid this error.

……

…… [1]

[Total: 16]

4 Planning

The enthalpy change of neutralisation, ΔH_{neut}, of ethanedioic acid, $H_2C_2O_4$, with sodium hydroxide, NaOH, is the molar enthalpy change for the reaction below.

$$\tfrac{1}{2}\ H_2C_2O_4(aq) + NaOH(aq) \rightarrow \tfrac{1}{2}\ Na_2C_2O_4(aq) + H_2O(l) \qquad \Delta H_{neut}$$

(a) Assume you are given the following solutions:

- 2.00 mol dm^{-3} NaOH;
- $H_2C_2O_4$ of approximately the same concentration.

The two solutions are mixed together to give a total volume of 50 cm^3.

Predict the volume of $H_2C_2O_4$ and of NaOH to be used such that both react completely. Show your working clearly and give your answers to the **nearest whole number**.

[1]

A series of experiments can be carried out using different volumes of the two solutions. In each experiment, the total volume of the two solutions is to be kept at 50 cm^3 and the increase in temperature, ΔT, is to be determined.

A graph of ΔT against *volume of* $H_2C_2O_4$ can then be used to determine the concentration of the $H_2C_2O_4$ solution and ΔH_{neut}.

(b) Plan a procedure to collect sufficient data to allow a graph of ΔT against *volume of* $H_2C_2O_4$ to be drawn.

You may assume that you are provided with:

- 2.00 mol dm^{-3} NaOH,
- $H_2C_2O_4$ solution of approximately the same concentration,
- the equipment normally found in a school laboratory.

Your plan should contain the following:

- a table to show the different volumes of $H_2C_2O_4$ and NaOH to be used and the temperature measurements to be recorded,
- essential experimental details including the apparatus you would use,
- a description of how you would minimise heat loss.

You may assume that the $H_2C_2O_4$ and NaOH solutions have the same temperature.

M1	
M2	
M3	
M4	
M5	

[5]

(c) Sketch on Fig. 4.1 the graph you would expect to obtain from your results. Label the axes including units.

Fig. 4.1

[2]

(d) Write calculation steps to show how you would use this graph to determine

- the concentration of the $H_2C_2O_4$ solution, in mol dm^{-3},
- ΔH_{neut}.

You may assume the following:

- specific heat capacity of all solutions is 4.18 J g^{-1} °C^{-1},
- density of all solutions is 1.00 g cm^{-3}.

[2]

[Total: 10]

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 aqueous 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)

(c) Test 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

(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