Name:		**Class:**	

ST ANDREW’S JUNIOR COLLEGE

JC2 PRELIMINARY EXAMINATION

CHEMISTRY **9729/04**

Paper 4 Practical **14 August 2023**

2 hours 30 minutes

Additional Materials: Qualitative Analysis Notes

Shift
Laboratory

READ THESE INSTRUCTIONS FIRST.

Write your name and class on all the work you hand in.
Give details of the practical shift and laboratory in the boxes provided above.
Write in dark blue or black pen.
You may use a soft pencil for any diagrams or graphs.
Do not use staples, paper clips, highlighters, 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.

The number of marks is given in the brackets [ ] at the end of each question or part question.

For Examiner’s Use	
1	**14**
2	**15**
3	**13**
4	**13**
Total	**55**

This document consists of **26** printed pages including this page.

1. Determination of *x* in hydrated copper(II) sulfate, $CuSO_4 \bullet xH_2O$ by titrimetric

analysis

The formula of hydrated copper(II) sulfate is $CuSO_4 \bullet \boldsymbol{x}H_2O$, where ***x*** represents the number of moles of water of crystallisation in one mole of the compound.

You are to determine the value of ***x*** by titrating a solution of iodine, I_2, with a solution of thiosulfate ions, $S_2O_3^{2-}$. The iodine is formed by the reaction of copper(II) ions, Cu^{2+}, with iodide ions, I^-.

$$2Cu^{2+} + 4I^- \rightarrow 2CuI + I_2$$

$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

You are provided with the following.

FA 1 is hydrated copper(II) sulfate, $CuSO_4 \bullet \boldsymbol{x}H_2O$.
FA 2 is 0.050 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 3 is 1.0 mol dm^{-3} potassium iodide, KI.

(a) Preparation of hydrated copper(II) sulfate, FA 4

1. Weigh accurately about 3.50 g of **FA 1** in a clean and dry weighing bottle. Record the mass of **FA 1** used on page 4.
2. Transfer **FA 1** into a 100 cm^3 beaker. Add approximately 80 cm^3 of deionised water to **FA 1** in the beaker and stir until the salt has dissolved. Rinse the weighing bottle with deionised water several times, adding each rinsing to the beaker.
3. Pour the contents carefully into a 250 cm^3 volumetric flask. Rinse the beaker with deionised water and add these washings to the flask.
4. Fill the flask to the mark with deionised water and shake to ensure thorough mixing.
5. Label the solution **FA 4**.

(b) (i) Titration of iodine against FA 2

1. Fill a burette with **FA 2**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 4** into a conical flask.
3. Use a measuring cylinder to add approximately 7.0 cm^3 of **FA 3** into the same conical flask. This will form a white precipitate in a brown solution.
4. Titrate this mixture with **FA 2**. When the brown solution becomes pale, add 1.0 cm^3 of starch indicator. The mixture will turn a blue-black colour.
5. Continue titrating this mixture until the blue-black colour just disappears to leave behind the white precipitate. This is the end-point of the titration.
6. Repeat steps 2 to 5 until consistent results are obtained.

Results

Mass of **FA 1** used = ……………..

☐ M1

☐ M2

☐ M3

☐ M4

[4]

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

☐ M5

volume of **FA 2** = …………………............ cm^3 **[1]**

(c) **(i)** Calculate the amount, in mol, of thiosulfate ions that were present in the volume of **FA 2** calculated in **(b)(ii)**.

☐ M6

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amount of $S_2O_3^{2-}$ = ……………….................. mol **[1]**

(ii) Use your answer to **(c)(i)** to obtain the amount, in mol, of copper(II) ions present in 25.0 cm^3 of **FA 4**.

amount of copper(II) ions in 25.0 cm^3 of **FA 4** = ……................. mol **[1]**

(iii) Determine the concentration, in mol dm^{-3}, of copper(II) ions in **FA 4**.

concentration of copper(II) ions in **FA 4** = ……............. mol dm^{-3} **[1]**

(iv) Use your answer to **(c)(iii)** to calculate the M_r of $CuSO_4 \bullet \boldsymbol{x}H_2O$.

M_r of $CuSO_4 \bullet \boldsymbol{x}H_2O$ = ……………………........................

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☐

Hence, deduce the value of ***x***. Show your working.
[A_r: Cu, 63.5; O, 16.0; H, 1.0; S, 32.1]

x = ……………………......................... **[3]**

(d) State and explain how the use of a measuring cylinder to measure the volume of **FA 3** in step 3 of **(b)(i)** will affect the accuracy of the titration result. **[1]**

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(e) **(i)** When titrating iodine against **FA 2**, a student recorded that 23.40 cm^3 of **FA 2** was used. However, the actual volume of **FA 2** added was 23.50 cm^3.

Considering that the maximum error in a single burette reading is ±0.05 cm^3, explain why the student's titre volume is accurate. **[1]**

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(ii) Student **B** suggested improving the accuracy of the titration results by changing the concentration of **FA 2** to 0.040 mol dm^{-3}.

State and explain if the student's suggestion would make the titration results more accurate. **[1]**

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[Total: 14]

2. Determination of the kinetics of a redox reaction

In acidic solutions, iron(III) ions are reduced by iodide ions to form iron(II) ions. The iodide ions are oxidised to iodine.

$$2Fe^{3+}(aq) + 2I^{-}(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$$

The rate of this reaction can be investigated by using starch indicator, which turns blue-black in the presence of iodine. Sodium thiosulfate is added to the reaction mixture to react with iodine as it is formed. The blue-black colour is seen when all the thiosulfate has reacted.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^{-}(aq) + S_4O_6^{2-}(aq)$$

You will investigate how the rate of reaction is affected by changing the concentration of the iodide ions.

FA 5 is 0.0500 mol dm^{-3} potassium iodide, KI.
FA 6 is 0.0500 mol dm^{-3} acidified iron(III) chloride, $FeCl_3$.
FA 7 is 0.00500 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.

(a) Prepare a table on page 10 for your results to be recorded to an appropriate level of precision. You will need to include the volume of **FA 5**, volume of water, reaction time and the rate of reaction for each of the five experiments.

Note: In each of these experiments, you will need to place the beaker on a printed page on page 1 of the insert. You will view the page by looking vertically down through the mixture. You will stop the stopwatch when you can no longer see the prints on the printed page.

Experiment 1

1. Fill a burette with **FA 5**.
2. Transfer 20.00 cm^3 of **FA 5** into a 100 cm^3 beaker.
3. Use a 25.0 cm^3 measuring cylinder to measure 20.0 cm^3 of **FA 7** and add to the same 100 cm^3 beaker.
4. Use a 10.0 cm^3 measuring cylinder to measure 5.0 cm^3 of starch indicator and add to the same 100 cm^3 beaker.
5. Use another 10.0 cm^3 measuring cylinder to measure 10.0 cm^3 of **FA 6**.
6. Pour the **FA 6** into the same 100 cm^3 beaker and start timing immediately. Ignore the initial purple colour observed.
7. Stir once and place the beaker on the printed page on page 1 of the insert.
8. Stop timing as soon as the solution turns blue-black and obscures the prints on the printed page.
9. Record this reaction time to the nearest second.
10. Wash the beaker and dry it with a paper towel.

Experiment 2

1. Fill a second burette with deionised water.
2. Transfer 10.00 cm^3 of **FA 5** into a 100 cm^3 beaker.
3. Transfer 10.00 cm^3 of deionised water into the beaker containing **FA 5**.
4. Use a 25.0 cm^3 measuring cylinder to measure 20.0 cm^3 of **FA 7** and add to the same 100 cm^3 beaker.
5. Use a 10.0 cm^3 measuring cylinder to measure 5.0 cm^3 of starch indicator and add to the same 100 cm^3 beaker.
6. Use another 10.0 cm^3 measuring cylinder to measure 10.0 cm^3 of **FA 6**.
7. Pour the **FA 6** to the same 100 cm^3 beaker and start timing immediately. Ignore the initial purple colour observed.
8. Stir once and place the beaker on the printed page on page 1 of the insert.
9. Stop timing as soon as the solution turns blue-black and obscures the prints on the printed page.
10. Record this reaction time to the nearest second.
11. Wash the beaker and dry it with a paper towel.

For Examiner's Use

Experiments 3–5

1. Carry out three further experiments to investigate how the reaction time

changes with different volumes of potassium iodide, **FA 5**. The combined volume of **FA 5** and deionsied water must always be 20.00 cm^3. Do not use a volume of **FA 5** that is less than 6.00 cm^3.

Results

The rate of reaction can be calculated as shown:

$$\text{Rate} = \frac{1000}{Reaction\ Time}$$

[5]

(b) **(i)** Calculate the amount, in mol, of the thiosulfate ions, $S_2O_3^{2-}$, used in each experiment.

amount of $S_2O_3^{2-}$ = ……………………… mol **[1]**

For Examiner's Use

(ii) Calculate the amount, in mol, of the iodide ions, I^-, that were used to produce the amount of iodine that reacted with the amount of $S_2O_3^{2-}$ in **(b)(i)**.

☐

amount of I^- = ……………………… mol **[1]**

(iii) Using your answer to **(b)(ii)**, calculate the concentration of I^- up to the time of appearance of the blue colour.

concentration of I^- = ……………………… **[2]**

For Examiner's Use

(c) Plot a graph of rate on the y-axis against volume of **FA 5** on the x-axis. Draw the best-fit straight line taking into account all of your plotted points.

☐ M24

☐ M25

☐

[3]

For Examiner's Use

(d) Using your graph, deduce the effect of changing the concentration of KI on the rate of the reaction between Fe^{3+}(aq) and I^-(aq). **[1]**

………………………………………………………………………………………..

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(e) A student carries out the same investigation as in **(a)** but the solutions are mixed in a different order. The student places **FA 5** and an appropriate volume of deionised water in one beaker and all the other reactants in a second beaker. The student then transfers the mixture from the second beaker to the first beaker and starts timing.

Explain which method is a better method.

You may use the data in the table below to explain your answer.

electrode reaction	**E^o / V**
$S_4O_6^{2-} + 2e^- \rightleftharpoons 2S_2O_3^{2-}$	+0.09
$I_2 + 2e^- \rightleftharpoons 2I^-$	+0.54
$Fe^{3+} + e^- \rightleftharpoons Fe^{2+}$	+0.77

[2]

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[Total: 15]

3. Qualitative Analysis

In this question you will deduce the identities of the following organic and inorganic compounds.

- ethanol, CH_3CH_2OH
- methanoic acid, HCOOH
- metal halide

You are provided with aqueous samples of three of these compounds, **FA 8, FA 9** and **FA 10**.

Perform the tests described in Table 3.1 on page 15. Some of the observations have been completed for you. There is no need to carry out those tests. Record your observations in Table 3.1.

You will also plan and conduct your own experiments in Table 3.2 on page 18.

Using the observations in Table 3.1 and Table 3.2, you will then deduce the identities of **FA 8, FA 9** and **FA 10.**

Test and identify any gases evolved. If there is no observable change, write **no observable change**.

You are advised to carry out the experiments on each unknown compound one at a time.

Table 3.1

Test		*Observations*		
		FA 8	**FA 9**	**FA 10**
(a)	Test the unknown using Universal Indicator paper.			Universal Indicator paper turned light green. pH is 7.
(b)	To 1 cm depth of the unknown in a test-tube, add 1 cm depth of sodium carbonate.			No effervescence seen.
(c)	To 1 cm depth of the unknown in a test-tube, add 1 cm depth of dilute sulfuric acid, followed by 3 drops of potassium manganate(VII) solution. Place the test-tube in the hot			

For Examiner's Use

☐ M30

☐ M31

	water bath for 5 minutes. Test and determine if any gas is evolved using a moist blue litmus paper.			
(d)	To 1 cm depth of aqueous iodine, add aqueous sodium hydroxide dropwise until a permanent yellow colour is obtained. Then add 5 drops of the unknown into the same test-tube. Place the test-tube in the hot water bath for 1 minute.			

For Examiner's Use

[4]

(e) **Identifying unknown metal halide**

(i) From your observations in Table 3.1, identify and explain which of the unknown compounds is the metal halide.

FA ………

explanation: ……………………………………………………………

…………………………………………………………………………..

…………………………………………………………………………..

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

(ii) Suggest the reagent and carry out the test to confirm the identity of the halide in the metal halide.

reagent: ……………………………………………………………...…..

observations: ……………………………………………………………

…………………………………………………………………………..

FA …….. contains ……………. anion. **[2]**

(iii) In Table 3.2 below, describe two tests that will allow you to identify the cation in the metal halide. Perform the tests and record the observations in the same table. Use only the bench reagents provided.

Table 3.2

Test	*Observations*
	FA ……..

The cation in metal halide is ………… **[3]**

(iv) The nature of the cation of the metal halide in aqueous solution accounts for its observations in test **(a)** and **(b)** in Table 3.1. Describe and explain the nature of the cation by means of an equation.

equation: ………………………………………………………………….

☐ M40

explanation: ……………………………………………………………...

☐ M41

…………………………………………………………………………...

[2]

…………………………………………………………………………

(f) **Identifying organic liquids**

From your observations in Table 3.1, identify which unknown is methanoic acid, HCOOH, and state the evidence.

FA ……..

evidence: ……………………………………………………...…………

☐ M42

……………………………………………………………………………...

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

[Total: 13]

4. **Planning: Determining the concentration of aqueous methanoic acid and the enthalpy change of neutralisation**

When aqueous methanoic acid, HCOOH (aq), is mixed with aqueous sodium hydroxide, NaOH (aq), the reaction releases heat causing a rise in the temperature of the solution.

$$\text{HCOOH (aq)} + \text{NaOH (aq)} \rightarrow \text{HCOONa (aq)} + \text{H}_2\text{O} (l) \qquad \Delta H_{\text{neut}} < 0$$

A series of experiments can be performed where increasing volumes of aqueous methanoic acid and decreasing volumes of aqueous sodium hydroxide are mixed and the temperature rise, ΔT, for each experiment is determined.

In each of the experiments using different volumes of aqueous methanoic acid and aqueous sodium hydroxide, the **total** volume has to be kept **constant**. Since the total volume of mixture remains the same, the temperature rise, ΔT, is a direct measure of the heat given out by the reaction. The maximum amount of heat is evolved when all the acid present is exactly neutralised by all the alkali.

The weighted average initial temperature, $T_{average,}$ of methanoic acid and sodium hydroxide may be calculated using the formula given below:

$$T_{average} = \frac{(V_{HCOOH}\ x\ T_{HCOOH}) + (V_{NaOH}\ x\ T_{NaOH})}{Total\ volume\ of\ the\ mixture}$$

Plotting a graph of ΔT against the volume of methanoic acid will give two straight lines of best–fit.

Extrapolation of the two straight lines will produce a point of intersection from which the concentration of aqueous methanoic acid and the enthalpy change of neutralisation, ΔH_{neut}, of the exothermic reaction between aqueous methanoic acid and aqueous sodium hydroxide can be determined.

The volume of aqueous methanoic acid used should be at least 20.00 cm^3 and the total volume of the reaction mixture should be kept constant at 60.00 cm^3 for all experiments.

For Examiner's Use

(a) Fill up Table 4.1 for the volume of methanoic acid and volume of sodium hydroxide to be used in the six experiments to plot a suitable Δ*T*–volume of methanoic acid graph.

Table 4.1

Experiment number	V_{HCOOH} / cm^3	V_{NaOH} / cm^3
1		
2		
3		
4		
5		
6		

[1]

(b) Plan an investigation to determine the concentration of methanoic acid and the enthalpy change of neutralisation, ΔH_{neut}, of the exothermic reaction between aqueous methanoic acid and aqueous sodium hydroxide.

You may assume that you are provided with:

- methanoic acid solution of approximate concentration 1.8 mol dm^{-3},
- 1.5 mol dm^{-3} aqueous sodium hydroxide,
- the equipment normally found in a school or college laboratory.

In your plan you should include brief details of:

- the apparatus you would use
- the quantities you would use
- the procedure you would follow
- the measurements you would make to allow a suitable Δ*T*–volume of methanoic acid graph to be drawn
- how the data measured would be used to determine values needed for the plotting of the graph
- how you would ensure that an **accurate** and **reliable** value of the concentration of methanoic acid and ΔH_{neut} is obtained.

[5]

For Examiner's Use

☐ M44

☐ M45

☐ M46

☐ M47

☐ M48

(c) Sketch on Fig 4.1 the graph you would expect to obtain from your results. Indicate clearly on your sketch how you would determine:

- $\boldsymbol{V}_{\text{neut}}$, the volume of HCOOH (aq) needed to just completely neutralise (60 – $\boldsymbol{V}_{\text{neut}}$) cm^3 of NaOH (aq)
- $\Delta \boldsymbol{T}_{\text{max}}$, the maximum temperature rise when the stoichiometric amount of HCOOH (aq) and NaOH (aq) reacted. **[2]**

☐ M49

☐ M50

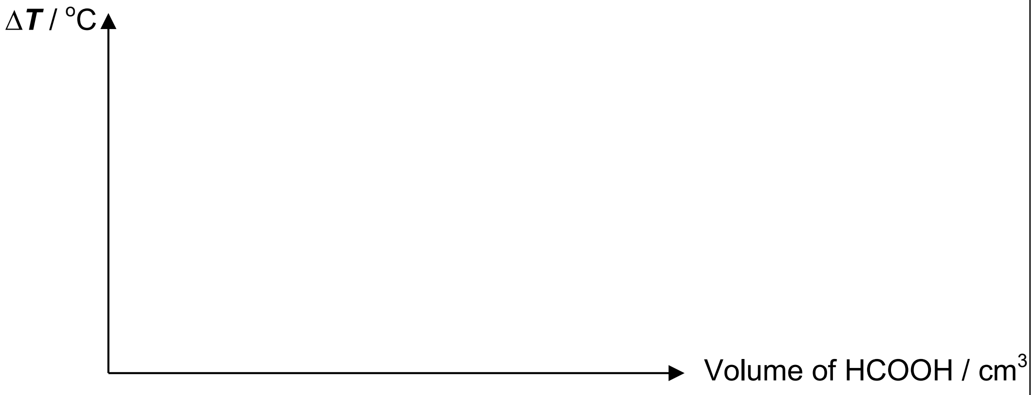

Fig 4.1

(d) **(i)** Given that the volume of methanoic acid is 27.50 cm^3 at $\Delta \boldsymbol{T}_{\text{max}}$, determine the concentration, in mol dm^{-3}, of HCOOH (aq). **[1]**

☐ M51

concentration of methanoic acid = …………………… mol dm^{-3}

(ii) Determine the enthalpy change of neutralisation, ΔH_{neut}, for this reaction. Express your answers in terms of ΔT_{max}.

You should assume that the specific heat capacity of the final solution is 4.18 J g^{-1} K^{-1} and its density is 1.00 g cm^{-3}. **[2]**

☐ M52

☐ M53

ΔH_{neut} = …………………… kJ mol^{-1}

(e) A similar exothermic reaction takes place between a monobasic strong acid, aqueous HX, and aqueous sodium hydroxide.

Explain how the following terms would differ from the values obtained in **(c)** when the same experiment described in **(b)** is repeated using a monobasic strong acid, aqueous HX, of the same concentration as aqueous HCOOH.

- $\boldsymbol{V}_{neut}$, the volume HX (aq) needed to just completely neutralise (60 – $\boldsymbol{V}_{neut}$) cm^3 of NaOH (aq)
- $\Delta \boldsymbol{T}_{max}$, the maximum temperature rise when the stoichiometric amount of HX (aq) and NaOH (aq) reacted. **[2]**

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☐ M54

☐ M55

[Total: 13]

[Turn Over

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 → (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 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