Name:		Class:	

ST ANDREW'S JUNIOR COLLEGE

JC2 PRELIMINARY EXAMINATION

CHEMISTRY **9729/04**

Paper 4 Practical **15 August 2024**

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	/13
2	/17
3	/15
4	/10
Total	/55

This document consists of **18** printed pages including this page.

Answer **all** the questions in the spaces provided.

1 Identification of unknown cation, anion and acids

FA 1 is a salt which contains a cation and an anion.
FA 2 is an aqueous mixture of two acids. The possible acids are hydrochloric acid, nitric acid and sulfuric acid.

You will perform tests to identify the ions in **FA 1** and determine which acids are present in **FA 2**.

Test and identify any gases evolved, **unless otherwise stated**.

If there is no observable change, write **no observable change**.

(a) (i) Carry out the following tests and record your observations in Table 1.1.

Table 1.1

	tests	observations
1	Add approximately one spatula of **FA 1** in a boiling tube and heat strongly for about 1 minute, then leave to cool. **Do not test for gas.**	
2	To an approximately 2 cm depth of **FA 2** in a test tube, carefully add approximately one spatula of **FA 1**. **Keep this solution for use in 1(b)(i).**	

[4]

(ii) Identify the anion in **FA 1** and write an ionic equation to explain how you deduced its identity, using your observations in Table 1.1.

……

…… **[1]**

(b) (i) Devise and perform a series of simple tests on the solution from Test 2 in **(a)(i)** to identify the cation in **FA 1**. Your tests should be based on the **Qualitative Analysis Notes on pages 17 to 18** and should only use the bench reagents provided**.**
Record your tests and observations in the space below.

[2]

(ii) Use your observations in **(b)(i)** to identify the cation in **FA 1**.

…..…… **[1]**

(c) (i) Devise and perform a series of simple tests to identify the acids present in **FA 2**. Your tests should be based on the **Qualitative Analysis Notes on pages 17 to 18** and should only use the bench reagents provided**.**
Record your tests and observations in the space below.
Any test requiring heating MUST be performed in a boiling tube.

[4]

(ii) State the formulae of the acids present in **FA 2**.

…….. **[1]**

[Total: 13]

2 Determination of the identity of a straight-chain carboxylic acid

Compound **E** is a straight-chain monobasic carboxylic acid. It is reacted with excess alkali and the unreacted alkali is then titrated with an acid.

FA 3 was prepared as follows:

- 200 cm^3 of 0.30 mol dm^{-3} aqueous sodium hydroxide was transferred into a beaker.
- 2.5 g of **E** was added.
- The solution was heated for 1 minute to ensure complete reaction.

To determine the identity of compound **E**, you will first perform a dilution of hydrochloric acid, **FA 4**, and titrate this with **FA 3**.

FA 4 is 2.00 mol dm^{-3} hydrochloric acid, HC*l*. You will also use **FA 4** for Question 3.
Solution S is screened methyl orange indicator.

(a) (i) Dilution of FA 4

1. Use a pipette to transfer 25.0 cm^3 of **FA 4** into a 250 cm^3 volumetric flask.
2. Make up the contents in the volumetric flask to the mark with deionised water.
3. Stopper the flask and mix thoroughly to obtain a homogeneous solution. Label this diluted acid as **FA 5**.
4. Wash the pipette thoroughly before moving on to the next set of instructions for titration.
5. **Do not discard FA 4. Keep the remaining FA 4 solution for Question 3.**

(ii) Titration of FA 3 against FA 5 using solution S

1. Fill the burette with **FA 5**.
2. Use a pipette to transfer 25.0 cm^3 of **FA 3** into a conical flask.
3. Add a few drops of **solution S** into the conical flask.
4. Run **FA 5** from the burette into the conical flask. The end-point is reached when the solution changes from green to grey. If the solution becomes violet, you have passed the end-point.
5. Record your titration results to an appropriate level of precision in the space on the next page.
6. Repeat steps 2 to 5 until consistent results are obtained.

Titration results

[2]

(iii) From your titrations, obtain a suitable volume of **FA 5** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 5** = ……. **[3]**

(b) (i) Calculate the amount, in moles, of sodium hydroxide added to compound **E** during the preparation of **FA 3**.

amount of NaOH added to **E** = ……. **[1]**

(ii) Use your titration results to calculate the amount, in moles, of sodium hydroxide in the beaker after reaction with compound **E** during the preparation of **FA 3**. Hence, calculate the amount, in moles, of sodium hydroxide that reacted with compound **E**.

amount of NaOH in the beaker after reaction with **E** = ……..

amount of NaOH that reacted with **E** = …….. **[3]**

(iii) Use your answer to **(b)(ii)** to calculate the M_r of **E**. Record your answer to 1 decimal place.

Hence, deduce the identity of **E**, showing your working clearly.
[A_r: C, 12.0; H, 1.0; O, 16.0]

M_r of **E** = ……..

Identity of **E** = …….. **[3]**

(c) **(i)** Suggest a reason why a dilution of **FA 4** was necessary before titrating it against **FA 3**.

……...

……... **[1]**

(ii) Explain why it was necessary to wash the pipette thoroughly after dilution of **FA 4** and what effect failing to do it would have on the titre value.

……...

……... **[2]**

(d) Given that the errors (uncertainties) associated with each reading using a volumetric flask and pipette are $\pm$ 0.15 cm^3 and $\pm$ 0.03 cm^3 respectively, calculate the maximum total percentage error (uncertainty) from the apparatus in a titration.

maximum total percentage error = …… % **[2]**

[Total: 17]

3 Determining temperature change of reaction to identify A_r of unknown element

A metal carbonate, XCO_3, reacts with hydrochloric acid (in excess) according to the following equation.

$$XCO_3 + 2HCl \rightarrow XCl_2 + CO_2 + H_2O \qquad \Delta H = -59.5 \text{ kJ mol}^{-1}$$

In this experiment, you will measure the temperature of hydrochloric acid in a Styrofoam cup at regular time intervals, before and after XCO_3 is added.

You will analyse your results graphically to obtain an accurate value for the temperature change caused by the reaction and use this value to calculate the heat change, q, for the experiment, and hence determine the relative atomic mass, A_r, of the metal X.

You are provided with:
FA 4, 2.00 mol dm^{-3} hydrochloric acid, HCl from Question 2;
FA 6, solid XCO_3 (placed beside the communal mass balances around the lab)

(a) In the space provided on page 10, prepare tables in which to record for your experiment:

- all weighings to an appropriate level of precision;
- all values of temperature, T, to an appropriate level of precision;
- all values of time, t, recorded to 0.5 min.

It is important that you measure each temperature at the specified time.

Procedure

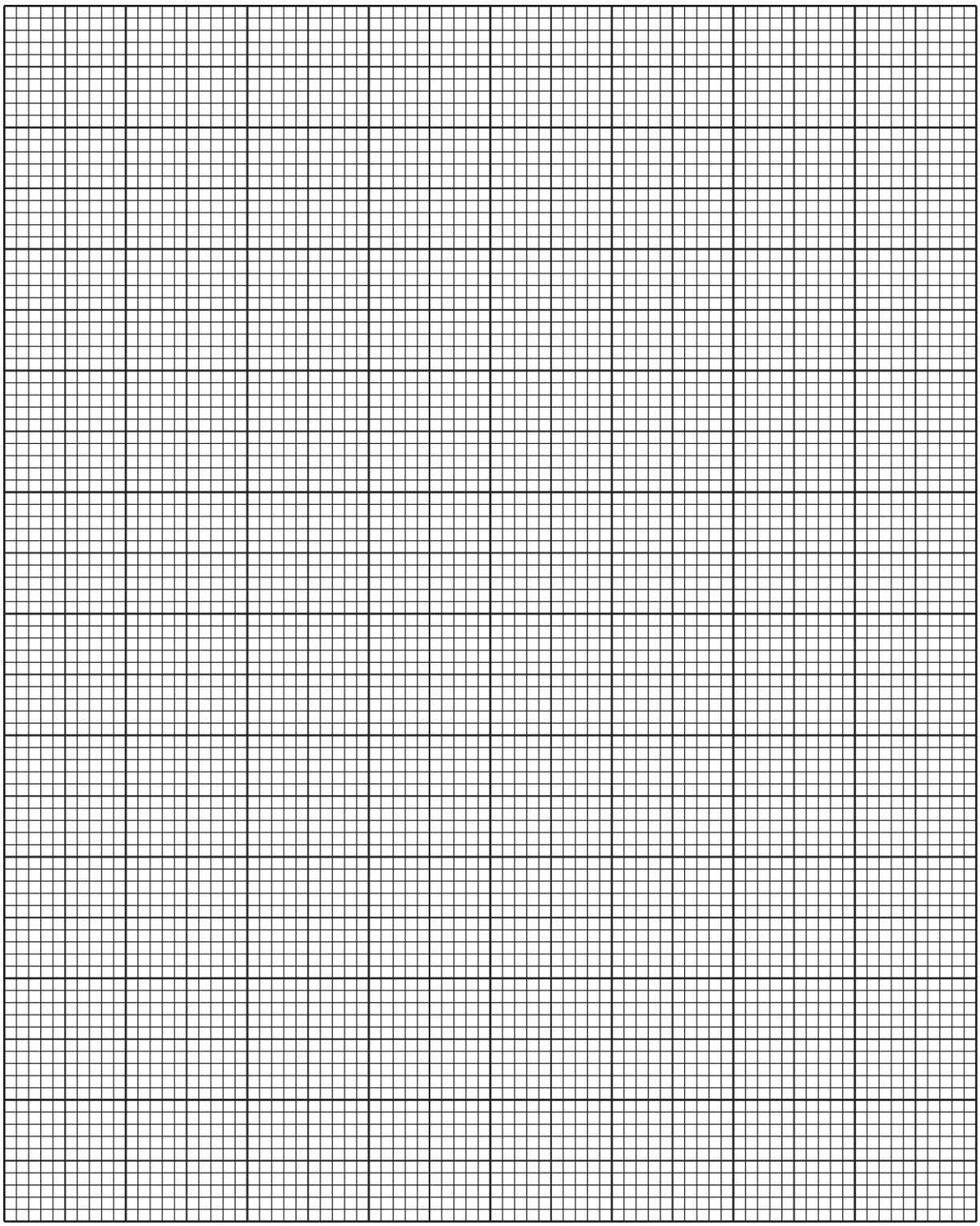
1. Weigh accurately about 3.20 g of **FA 6** into a clean and dry weighing bottle. Record the relevant masses in your table on page 10.
2. Place one Styrofoam cup inside another Styrofoam cup and place both in a 250 cm^3 beaker.
3. Use a 50.0 cm^3 measuring cylinder to transfer 50.0 cm^3 of **FA 4** into the Styrofoam cup.
4. Place the plastic lid with a hole in the centre on the cup and insert the thermometer through the lid. Carefully stir the **FA 4** in the Styrofoam cup with the thermometer. Read and record the temperature, T. This is the temperature at t = 0.0 min.
5. Start the stopwatch. Read and record the temperature at t = 0.5 min and t = 1.0 min.
6. At t = 1.5 min, lift the lid and the thermometer and **carefully** add **FA 6 slowly** into the acid. Close the lid and stir the mixture thoroughly, but **do not** record T.
7. Continue to stir the mixture. Read and record T at t = 2.0 min, and every 0.5 min until t = 4.0 min, and subsequently every minute until t = 8.0 min.
8. Reweigh the weighing bottle and record the mass of **FA 6** used.

Results

[4]

(b) Graph Plotting

1. Plot a graph of temperature, T, on the y-axis against time, t, on the x-axis, on the grid on page 11**.**
2. Draw two straight lines of best fit; one through the points before t = 1.5 min; the second through the points after temperature of the mixture has started to fall. Extrapolate (extend) both lines to t = 1.5 min.
3. From the graph, read the minimum temperature, T_{min}, and the maximum temperature, T_{max}, at t = 1.5 min. Record these values in the spaces provided below the graph grid.
4. Deduce the temperature change, ΔT, at t = 1.5 min.



T_{min} at t = 1.5 min = ...°C

T_{max} at t = 1.5 min = ...°C

ΔT at t = 1.5 min = ...°C **[4]**

(c) **(i)** Calculate the heat change, q, for your experiment. Assume that the specific heat capacity of the reaction mixture is 4.2 J g^{-1} K^{-1} and that the density of the reaction mixture is 1.00 g cm^{-3}.

[1]

(ii) Calculate the amount, in moles, of **FA 6** that reacted with hydrochloric acid.

[1]

(iii) Calculate the relative atomic mass, A_r, of the metal X. Leave your answer to 1 decimal place.
[A_r: C, 12.0; O, 16.0]

[3]

(d) **(i)** Suggest why a direct titration of XCO_3 with HC*l* is not recommended even though volumetric analysis generally yields more accurate results.

……

…… **[1]**

(ii) A student repeated the procedure in **(a)** but used 2.00 mol dm^{-3} sulfuric acid instead of **FA 4** in Step 3. Predict the effect of this on ΔT in **(b).**

……

…… **[1]**

[Total: 15]

4 Planning

There is a direct relationship between the rate of chemical reactions and the temperature of the reaction mixture. This mathematical relationship is

$$\log_{10} (\text{rate of reaction}) = \frac{-E_a}{19T}$$

where:

- E_a = activation energy of the reaction
- T = temperature (in Kelvin)
- rate of reaction = reciprocal of the time taken (1/time) in seconds.

One reaction that can be used to investigate this relationship is the reaction between dilute hydrochloric acid and aqueous sodium thiosulfate.

$$Na_2S_2O_3\,(aq) + 2HCl\,(aq) \rightarrow 2NaCl\,(aq) + S\,(s) + SO_2\,(g) + H_2O\,(l)$$

The activation energy, E_a for the reaction may be calculated by measuring the time taken for the solution to be opaque due to the solid sulfur produced at different temperatures.

You may assume that you are provided with:
0.10 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$;
0.20 mol dm^{-3} hydrochloric acid, HCl
Equipment normally found in a school or college laboratory.

(a) State and explain the type of reaction between sodium thiosulfate and hydrochloric acid.

……..

…….. **[1]**

(b) Plan a procedure to collect sufficient data to plot a graph of $\log_{10}(1/t)$ against $1/T$ where t is the time taken for the reaction mixture to turn opaque and T is the reaction temperature in Kelvin.
The reaction temperature for each reaction should be between 40 °C and 70 °C.
You may use 20.0 cm^3 each of the provided solutions of sodium thiosulfate and hydrochloric acid for each experiment.

In your plan, you should include details of:

- the apparatus you would use
- the procedure you would follow
- the measurements you would take to obtain the necessary readings required for graphical analysis

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

……..

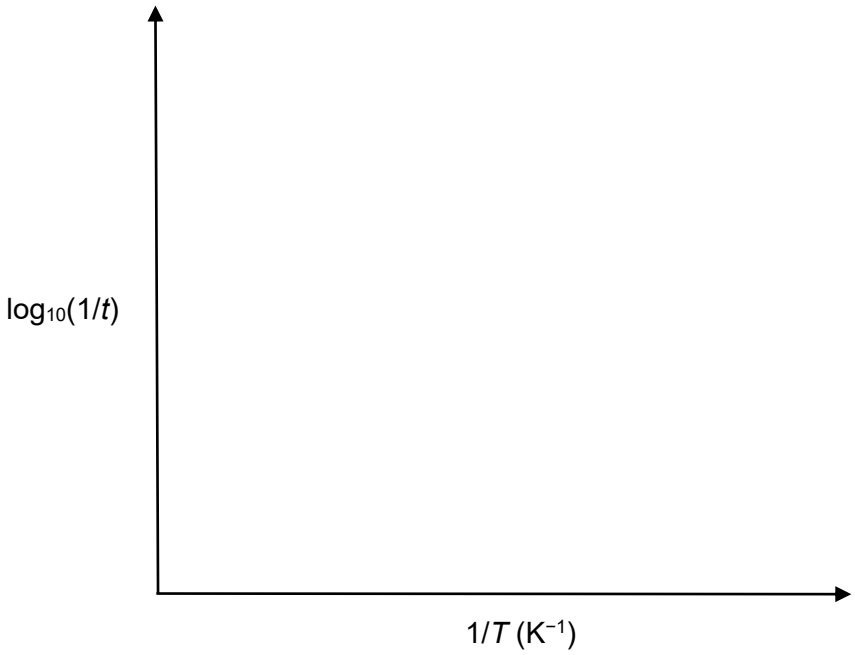
……..

……..

……..

……..[6]

(c) Sketch the graph that you would expect to obtain on the axes below and describe how you would use your graph to determine the value of E_a.



……...

……...

……... **[2]**

(d) A student carried out your plan in **(b)** and found that the time taken for the solution to be opaque is too short for each experiment, leading to a high percentage error. Describe how you would modify your plan to address this concern.

……...

……...

……... **[1]**

[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 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 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