Name:	C	lass:				
I	ST ANDREW'S JUNIOR COL	LEGE				
	JC2 PRELIMINARY EXAMINA	ATION				
CHEMIST	RY			9729/04		
Paper 4 Pr	ractical		15 2 hour	August 2024		
Additional	Materials: Qualitative Analysis Notes			Shift		
READ TH	ESE INSTRUCTIONS FIRST.					
Write your	name and class on all the work you hand in.		La	Laboratory		
Give detail	s of the practical shift and laboratory in the boxes			-		
provided a	provided above.					
Write in da	rk blue or black pen.		For Exam	niner's Use		
You may u	1					
Do not use		13				
fluid.	2					
				17		
Answer al l	questions in the spaces provided on the Question		3			
Paper.				15		
The use of	an approved scientific calculator is expected, where	•	4			
appropriate	appropriate.					
You may I	Total					
you do no	t use appropriate units.			55		
The number	er of marks is given in the brackets [] at the end of					
each question or part question.						
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.

	tests	observations		
1	Add approximately one spatula of FA 1	White solid turned yellow upon strong		
	in a boiling tube and heat strongly for	heating.		
	about 1 minute, then leave to cool.			
		Yellow solid turned white again upon cooling.		
		Note: Some students might have also		
		observed colourless droplets forming		
		on the top of the boiling tube.		
2	To an approximately 2 cm depth of FA	White solid dissolved to form		
	2 in a test tube, carefully add	colourless solution.		
	approximately one spatula of FA 1 .			
		Effervescence observed. Gas forms		
		white ppt with limewater.		
	Keep this solution for use in	Gas is <u>CO</u> 2.		
	1(b)(i).			

Table 1.1

(ii) Identify the anion in **FA1** and write an ionic equation to explain how you deduced its identity, using your observations in Table 1.1.

Anion is CO_3^{2-} $2H^++CO_3^{2-} \rightarrow CO_2 + H_2O$

(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.



- (ii) Use your observations in (b)(i) to identify the cation in FA 1.
 Zn²⁺ (ecf from observations)
 [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.

Tests	Observations
To 1 cm ³ of FA 2 in a test tube, add AgNO ₃ (aq), followed by NH ₃ (aq).	$\frac{\text{White ppt}}{\text{soluble in NH}_3}$ formed which was soluble in NH ₃ to form colourless solution.
To 1 cm ³ of FA 2 in a boiling tube, add 1 cm ³ of <u>NaOH(aq)</u> and place 1 piece of <u>AI foil</u> inside the boiling tube. Heat gently.	Gas evolved turns damp red litmus paper blue.
To 1 cm ³ of FA 2 in a test tube, add <u>Ba(NO₃)₂(aq).</u>	No ppt formed.

[4]

(ii) State the formulae of the acids present in FA 2. HCl and HNO_3

.....[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 cm³ 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 cm³ of **FA 4** into a 250 cm³ 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

Titration number	1	2
Final burette reading / cm ³	20.25	40.50
Initial burette reading / cm ³	0.00	20.25
Volume of FA 5 / HC/ used / cm ³	20.25	20.25

(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** used = $\frac{20.25 + 20.25}{2} = 20.25 \text{ cm}^3$

[3] volume of **FA 5** =

- (b) (i) Calculate the amount, in moles, of sodium hydroxide added to compound E during the preparation of FA 3.
 Initial amount of NaOH = 0.200 x 0.30 = 0.0600 mol
 - 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.

[HC/] in FA 5 = $2.00 / 10 = 0.200 \text{ mol dm}^{-3}$ Amount of HC/ used in titration = $0.200 \times 0.02025 = 0.00405 \text{ mol}$ = Amount of NaOH in 25.0 cm³ of FA 3 Amount of unreacted NaOH in 200.0 cm³ of FA 3 (beaker) = $0.00405 \times (200/25)$ = 0.0324 mol

Amount of NaOH that reacted with E = 0.06 - 0.0324 = 0.0276 mol (ecf)

(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] Amount of E = 0.0276 mol M_r of E = 2.5 / 0.0276 (ecf) = <u>90.6</u> (1 d.p.)

*M*_r of **E** =

 M_r of COOH = 45 M_r of C_xH_{2x+1} = 45.6 (ecf) x = 3 (approx.) E is butanoic acid (C₃H₇COOH) (must be straight-chain COOH)

Identity of **E** =

[3]

[3]

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



flask and pipette are ± 0.15 cm³ and ± 0.03 cm³ respectively, calculate the maximum total percentage error (uncertainty) from the apparatus in a titration. Percentage error in using volumetric flask = $\pm \frac{0.15}{250} \times 100\% = \pm 0.0600\%$ Percentage error in using the pipette = $\pm \frac{0.03}{25.0} \times 2 \times 100\% = \pm 0.120\%$ Percentage error in using the burette = $\pm \frac{0.05}{20.25} \times 2 \times 100\% = \pm 0.4938\%$ Maximum total percentage error from the apparatus = $\pm 0.06 + 0.12 + 0.4938 = \pm 0.794\%$ (3 sf)

[Total: 17]

[2]

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.

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

In this experiment, you will measure the temperature of hydrochloric acid in a Styrofoam cup at regular time intervals, before and after XCO₃ 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, HC*l* from Question 2;

FA 6, solid XCO₃ (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 cm³ beaker.
- 3. Use a 50.0 cm³ measuring cylinder to transfer 50.0 cm³ 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

Mass of empty weighing bottle / g	
Mass of FA 6 and weighing bottle / g	
Mass of emptied weighing bottle / g	
Mass of FA 6 used / g	3.20

Time, <i>t</i> / min	0.0	0.5	1.0	2.0	2.5	3.0	3.5	4.0	5.0	6.0	7.0	8.0
Temperature	30.	30.	30.	34.	34.	34.	34.	34.	34.	34.	34.	34.
/ °C	6	6	6	8	9	9	9	8	7	6	5	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.

[4]





		[4]
ΔT at <i>t</i> = 1.5 min =	+4.5	… °C
$T_{\rm max}$ at $t = 1.5$ min =	35.1	°C
T_{\min} at $t = 1.5 \min =$	30.6	°C

(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.

$$q = mc\Delta T$$

= 50 x 4.2 x 4.5 = 945 J [1]

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

 $\Delta H = -q/n$ Amount of FA 6 = $-q / \Delta H = -0.945 / -59.5 = 0.0159 \text{ mol} (3 \text{ s.f.})$

[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]

 A_r of XCO₃ = 3.20 / 0.0159 = 201.48 A_r of X = 201.48 - 12.0 - 48.0 = <u>141.5 (1 dp</u>)

XCO₃.

(d) (i) Suggest why a direct titration of XCO₃ with HC*l* is not recommended even though volumetric analysis generally yields more accurate results.

.....[1]

It is <u>difficult to determine the end-point</u> when XCO_3 is <u>insoluble</u>. Or The reaction between XCO_3 and HCl results in an <u>acid spray</u> which causes the <u>loss of XCO_3 or HCl</u>, leading to inaccurate results. Or The <u>colour of the indicator cannot be determined</u> when it is added to the solid

(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).

.....

<u>No effect</u>. XCO_3 is the limiting reagent so the <u>amount of XCO_3 reacted is the</u> <u>same</u>.

[Total: 15]

[3]

4 Planning

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

$$\log_{10}$$
 (rate of reaction) = $\frac{-E_a}{197}$

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, HC*l* Equipment normally found in a school or college laboratory.

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

<u>Disproportionation</u> of sulfur. The oxidation state of S changed from $\pm 2 \text{ in } S_2O_3^{2-}$ to 0 in S and to $\pm 4 \text{ in } SO_2$. [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 cm³ 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

Procedure [7 marks]:

- Measure 20.0 cm³ of sodium thiosulfate using a <u>25.0 cm³ measuring cylinder</u> (A) into a clean and dry <u>250 cm³ conical flask</u>. (A)
- 2. Measure 20.0 cm³ of HCl using another <u>25.0 cm³ measuring cylinder</u> (A).
- 3. Set up a water bath (A) that is thermostatically controlled (P) at 40 °C.
- 4. Place the conical flask containing sodium thiosulfate inside the water bath. (P)
- 5. Place a <u>thermometer</u> (A) inside the sodium thiosulfate solution to <u>ensure the</u> <u>temperature reaches 40 °C (P)</u>.
- 6. <u>Pour the HCl from the measuring cylinder</u> rapidly <u>into the conical flask</u>. (P) <u>Start</u> (P) the <u>stopwatch</u> (A).
- 7. Mix the contents thoroughly by swirling the flask. (P)
- Place the conical flask <u>on a white tile with a "X" / laminated sheet with printed</u> words / "X". (P)
- Stop (P) the stopwatch when the solution first becomes opaque / completely obscure the words on the printed page (P) and record the time taken (M) (in seconds).
- 10. Discard the reaction mixture inside the conical flask immediately down the sink. Wash the conical flask and let it stand upside down on a paper towel to dry.
- 11. <u>Repeat</u> Steps 1 to 8 <u>using temperatures of 50°C, 60°C and 70°C</u> (P) (M)
- (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 .



 $1/T (K^{-1})$

Correct sketch (straight line with negative gradient) – 1 mark

- 1. Gradient of the graph = $-E_a/19$
- 2. $E_a = -Gradient$ of the graph x 19

Correct description - 1 mark

[2]

[6]

(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.
 I would <u>dilute / lower the concentration of sodium thiosulfate or hydrochloric acid or both</u>. This will cause the rate of reaction to be slower and hence longer time [1] taken for S to be formed.

OR increase the total volume of the reaction mixture by adding deionised water.

[Total: 10]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	reaction with					
cation	NaOH(aq)	NH₃(aq)				
aluminium,	white ppt.	white ppt.				
A/ ³⁺ (aq)	soluble in excess	insoluble in excess				
ammonium, NH₄⁺(aq)	ammonia produced on heating	-				
barium, Ba²⁺(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), Cu²⁺(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), Fe³⁺(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), Mn2+(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, Zn²⁺(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, C <i>l</i> ⁻(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 NH₃(aq))
nitrate, NO₃⁻(aq)	NH_3 liberated on heating with $OH^-(aq)$ and A/ foil
nitrite, NO₂⁻(aq)	NH ₃ liberated on heating with OH [−] (aq) and A/ foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)
sulfate, SO₄²⁻(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, NH₃	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, I2	black solid / purple gas	brown	purple