

# EUNOIA JUNIOR COLLEGE

JC2 Preliminary Examination 2021 General Certificate of Education Advanced Level Higher 2

CANDIDATE NAME					
CIVICS GROUP	2	0	_	INDEX NUMBER	

# **CHEMISTRY**

9729/04

Paper 4 Practical

31 August 2021 2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials:

As listed in the Confidental Instructions

#### **READ THESE INSTRUCTIONS FIRST**

Write your name, civics group and registration number on 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 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.

Qualitative Analysis Notes are printed on pages 23 and 24.

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	
:		

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1	/ 22		
2	/ 22		
3	/ 11		
Total	/ 55		

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[Turn Over

# Answer all the questions in the spaces provided.

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# 1 Investigation on the behaviour of acids and bases in aqueous solution

- FA 1 is 1.00 mol dm<sup>-3</sup> sodium hydrogencarbonate, NaHCO<sub>3</sub>
- FA 2 is sodium hydroxide, NaOH, of concentration between 1.5 mol dm<sup>-3</sup> and 2.0 mol dm<sup>-3</sup>
- FA 3 is 2.00 mol dm<sup>-3</sup> sulfuric acid, H<sub>2</sub>SO<sub>4</sub>

According to the Arrhenius theory, an acid-base neutralisation reaction involves reacting together  $H^+(aq)$  and  $OH^-(aq)$  to produce water molecules.

$$H^{+}(aq) + OH^{-}(aq) \rightarrow H_2O(1)$$

$$\Delta H_{\text{neutralisation}} = -57.1 \text{ kJ mol}^{-1}$$

This task involves two different acid-base reactions.

The first reaction is between sodium hydrogencarbonate, FA 1, and sodium hydroxide, FA 2.

reaction 1 NaHCO<sub>3</sub>(aq) + NaOH(aq) 
$$\rightarrow$$
 Na<sub>2</sub>CO<sub>3</sub>(aq) + H<sub>2</sub>O( $l$ )

 $\Delta H_{\text{reaction 1}}$ 

The molar enthalpy change for **reaction 1**,  $\Delta H_{\text{reaction 1}}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with NaOH.

Instead of using an indicator to determine the endpoint, you will perform a thermometric titration to determine the equivalence point of the reaction. The equivalence point is that point where  $H^{\dagger}(aq)$  from the acid and  $OH^{\dagger}(aq)$  from the base are present in equal molar amounts.

The second reaction is between sodium hydrogencarbonate, FA 1, and sulfuric acid, FA 3.

reaction 2 NaHCO<sub>3</sub>(aq) + 
$$\frac{1}{2}$$
H<sub>2</sub>SO<sub>4</sub>(aq)  $\rightarrow \frac{1}{2}$ Na<sub>2</sub>SO<sub>4</sub>(aq) + H<sub>2</sub>O(1) + CO<sub>2</sub>(g)  $\Delta H_{\text{reaction 2}}$ 

The molar enthalpy change for **reaction 2**,  $\Delta H_{\text{reaction 2}}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with H<sub>2</sub>SO<sub>4</sub>.

In your first experiment, you will perform a thermometric titration by adding portions of FA 2 progressively to a known volume of FA 1. You will continue adding FA 2 until the equivalence point is reacted and passed. Throughout the experiment you will note and record the temperature of the mixture after each addition.

You will then analyse your results graphically in order to determine the

- titration volume at the equivalence point,  $V_{\text{equivalence}}$ ,
- maximum temperature change, ΔT<sub>maximum 1</sub>,
- molar enthalpy change, ΔH<sub>reaction 1</sub>, for reaction 1.

In your second experiment, you will mix together a given volume of **FA 1** with a suitable volume of **FA 3**.

You will then determine the

- maximum temperature change, ΔT<sub>maximum 2</sub>,
- molar enthalpy change, ΔH<sub>reaction 2</sub>, for reaction 2.

#### (a) The reaction between FA 1 and FA 2

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In this task you will need to record the maximum temperature of the reaction mixture when specified volumes of **FA 2** have been added. It is important that the volume of **FA 2** recorded is the total volume you have added up to that point when the temperature reading was made.

Note: If you overshoot on an addition, record the **actual** total volume of **FA 2** added up to that point.

In an appropriate format in the space provided on page 4, record all values of temperature, T, and each total volume of **FA 2** added.

- (i) 1. Fill a burette with FA 2.
  - 2. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
  - 3. Using a pipette, transfer 25.0 cm<sup>3</sup> of **FA 1** to the first Styrofoam cup.
  - 4. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature.
  - 5. From the burette, add 2.00 cm<sup>3</sup> of **FA 2** to the cup and stir the mixture thoroughly.
  - 6. Read and record the maximum temperature of the mixture, *T*, and the volume of **FA 2** added.
  - 7. Repeat points 5 and 6 until a total of 30.00 cm<sup>3</sup> of **FA 2** has been added. After each addition of **FA 2**, record the maximum temperature of the mixture and the total volume of **FA 2** added up to that point.

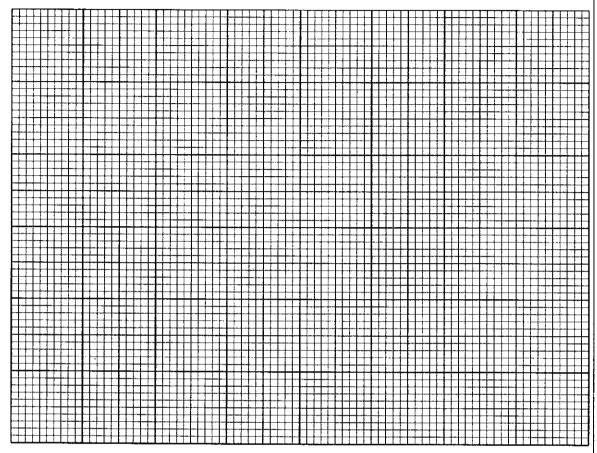
Results

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[2]

(ii) On the grid below, plot a graph of temperature, T(y-axis) against volume of FA 2 added (x-axis).

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[4]

- (iii) Draw two smooth lines of best fit.
  - The first best-fit line should be drawn using the plotted points where the temperature is rising.
  - The second best-fit line should be drawn using the plotted points where the temperature is falling.
  - Extrapolate these lines until they cross.

Note: Each line should have a shape best suited to its plotted points.

		<ul> <li>(iv) Determine from your graph</li> <li>the maximum temperature reached, T<sub>maximum</sub>,</li> <li>the maximum temperature change, ΔT<sub>maximum</sub> 1,</li> <li>the volume, V<sub>equivalence</sub>, of FA 2 needed to completely react with 25.0 cm<sup>3</sup> of FA 1.</li> </ul>
		Show on your graph how you obtained these values.
		Record these values in the spaces provided below.
		maximum temperature reached, $T_{\text{maximum}} = \dots$
		maximum temperature change, $\Delta T_{\text{maximum 1}} = \dots$
		volume of <b>FA 2</b> used, $V_{\text{equivalence}} = \dots$ [3]
(b)	ldea con	ally, the graph line <b>before</b> the equivalence point should be an <i>increasing</i> curve that caves downward (that is, gradient of the curve decreases).
	(i)	Explain why the graph line is increasing.
		[1]
	(ii)	Explain why the graph line concaves downward.
		[1]

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#### (c) The reaction between FA 1 and FA 3

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The molar enthalpy change for reaction 2,  $\Delta H_{\text{reaction2}}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with H<sub>2</sub>SO<sub>4</sub>.

In this task you will calculate a value for the molar enthalpy change,  $\Delta H_{\text{reaction 2}}$ . To do this, you will need to determine the maximum temperature change produced when measured volumes of **FA 1** and **FA 3** are mixed.

Upon considering the concentrations of **FA 1** and **FA 3**, 15 cm<sup>3</sup> of **FA 3** was chosen to add to 40 cm<sup>3</sup> of **FA 1**.

Note: You should be aware that the reaction mixture will produce considerable frothing.

(i) Explain why 15 cm³ of FA 3 was chosen to add to 40 cm³ of FA 1.

[1]

(ii) Identify the apparatus you intend to use to measure the volume of FA 1.

Explain your choice.

apparatus .....

explanation .....

[1]

(iii) In an appropriate format in the space provided below, record all values of measured temperature for this experiment.

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- Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
- Transfer 40 cm<sup>3</sup> of FA 1 into the first Styrofoam cup using the apparatus identified in 1(c)(ii).
- 3. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature,  $T_{\text{FA 1}}$ . This is the initial temperature of **FA 1**.
- 4. Wash and dry the thermometer.
- 5. Measure 15.0 cm $^3$  of **FA 3** using an appropriate measuring cylinder. Stir the **FA 3** solution in the measuring cylinder with the thermometer. Read and record its temperature,  $T_{\text{FA 3}}$ . This is the initial temperature of **FA 3**.
- Carefully add the contents of the measuring cylinder to the Styrofoam cup in small portions to avoid too much frothing.
- 7. Place the lid on the cup and insert the thermometer through the lid. Stir the mixture.
- 8. Continue to stir the mixture. Measure and record the temperature,  $T_{\rm mixture}$  that shows the maximum change from the initial temperature.

#### Results

[1]

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[2]

Cal	cula	tions
(d)	For a de	the purpose of these calculations, you should assume that the reaction mixture has ensity of 1.00 g cm <sup>-3</sup> and a specific heat capacity, $c$ , of 4.18 J g <sup>-1</sup> K <sup>-1</sup> .
	Use	your results from (a)(iv) to calculate
	(i)	the concentration of sodium hydroxide, [NaOH], in FA 2,
		· ·
		[NaOH] in <b>FA 2</b> =[1]
	(ii)	the heat change, $q$ , for the reaction occurring in (a)(i), and hence determine a value for the molar enthalpy change for reaction 1, $\Delta H_{\text{reaction 1}}$ .
		q =

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(e)	Use your results from (c)(iii) to calculate a value for the molar enthalpy change for
	reaction 2 AH
	reaction 2, Air reaction 2. To the formula given
	temperature, $T_{\text{average}}$ , of <b>FA 1</b> and <b>FA 3</b> may be calculated using the formula given
	below.

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$$\mathcal{T}_{\text{average}} = \frac{\left(V_{\text{FA}\,1} \times \mathcal{T}_{\text{FA}\,1}\right) + \left(V_{\text{FA}\,3} \times \mathcal{T}_{\text{FA}\,3}\right)}{\left(V_{\text{FA}\,1} + V_{\text{FA}\,3}\right)}$$

*q* = ......

$$\Delta H_{\text{reaction 2}} = \dots [1]$$

(f) Ionic equations for neutralisation, reaction 1, and reaction 2 are shown below.

$$H^{+} + OH^{-} \rightarrow H_{2}O$$
  $\Delta H_{\text{neutralisation}} = -57.1 \text{ kJ mol}^{-1}$   $HCO_{3}^{-} + OH^{-} \rightarrow CO_{3}^{2-} + H_{2}O$   $\Delta H_{\text{reaction 1}}$   $\Delta H_{\text{reaction 2}}$ 

Carbon dioxide may be removed from stale air by bubbling the air through aqueous alkali. An equation for this reaction is given below.

reaction 3 
$$2OH^{-}(aq) + CO_{2}(g) \rightarrow CO_{3}^{2-}(aq) + H_{2}O(l)$$
  $\Delta H_{reaction 3}$ 

Use your answers to **(d)(ii)** and **(e)**, together with the molar enthalpy change of neutralisation,  $\Delta H_{\text{neutralisation}}$ , to determine a value for the molar enthalpy change for this reaction,  $\Delta H_{\text{reaction 3}}$ .

$$\Delta H_{\text{reaction 3}} =$$
 [1]

(g) Another student planned a different thermometric titration experiment to determine the value of the titration volume at the equivalence point,  $V_{\text{equivalence}}$ , and the maximum temperature change,  $\Delta T_{\text{maximum 1}}$ , for **reaction 1**.

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In this other experiment, different volumes of **FA 1** and **FA 2** as shown in Table 1.1 are mixed, and the maximum temperature change,  $\Delta T_{\text{max}}$ , is determined for each mixture.

Table 1.1

experiment	1	2	3	4	5	6	7	8
V <sub>FA 2</sub> / cm <sup>3</sup>	10.00	15.00	20.00	25.00	30.00	35.00	40.00	45.00
V <sub>FA 1</sub> / cm <sup>3</sup>	45.00	40.00	35.00	30.00	25.00	20.00	15.00	10.00

 $\Delta T_{\rm max}$  is plotted against the volume of **FA 2** used,  $V_{\rm FA 2}$ .  $V_{\rm equivalence}$  and  $\Delta T_{\rm maximum 1}$  are determined from the intersection of the two straight lines drawn as shown in Fig. 1.1.

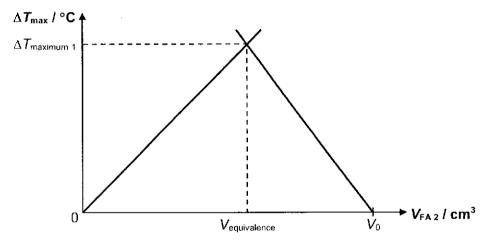


Fig. 1.1

(i) Suggest a value for  $V_0$  in Fig. 1.1.

3720		
$V_0 =$	 cm <sup>3</sup> [	1]

(ii) The student claimed that this method gives a more accurate value of  $V_{\text{equivalence}}$  and  $\Delta T_{\text{maximum 1}}$  compared to the method in **1(a)**.

Do you agree with the student? Explain.

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$\Delta T_{\sf maximum  1}$	Examiner's Use
[2]	
[Total: 22]	

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2 Determination of identity of the impurity in FA 4, the decomposition route of NaHCO<sub>3</sub>, the percentage purity of FA 4

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**Solid FA 4** is anhydrous sodium hydrogencarbonate, NaHCO<sub>3</sub>, of greater than 95% purity that has been contaminated with a sodium halide.

You are also provided with an aqueous solution of FA 4, labelled FA 4 solution.

- (a) You will devise and perform a simple test, based on the Qualitative Analysis Notes on pages 23–24, to identify the halide present in the **FA 4 solution**.
  - (i) Describe a test, using only the bench reagents provided, which will allow you to identify the halide impurity present in FA 4.

\_\_\_\_\_[1]

(ii) Perform the test you described in 2(a)(i) using the FA 4 solution provided.

Record your observations and hence deduce the identity	Of the Halide III Solid FA 4.
······	

identity of halide .....[2]

# (b) Thermal decomposition of NaHCO<sub>3</sub>

When NaHCO $_3$  is heated above 110 °C (but not heated to "red heat") it has been observed that both H $_2$ O(g) and CO $_2$ (g) are evolved, and that after this decomposition is complete, a white solid residue remains. Among possible reactions, the two following reactions seem the ones that are most likely to explain these facts:

reaction 4 2NaHCO<sub>3</sub>(s)  $\rightarrow$  Na<sub>2</sub>CO<sub>3</sub>(s) + H<sub>2</sub>O(g) + CO<sub>2</sub>(g)

reaction 5  $2NaHCO_3(s) \rightarrow Na_2O(s) + H_2O(g) + 2CO_2(g)$ 

Sodium halides are stable to heat.

In this experiment, solid  ${\sf FA~4}$  is heated gently in a boiling tube, over a luminous Bunsen flame, until  ${\sf all~NaHCO_3}$  has been decomposed. The data will be used to determine

- the percentage mass loss,
- whether the decomposition takes place via reaction 4 or reaction 5,
- the amount of CO<sub>2</sub> lost,
- the percentage purity of NaHCO₃ in FA 4

[Ar: Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

1.	Weigh accurately between 2.00 g and 2.50 g of <b>FA 4</b> in the boiling tube provided. Record your weighings in the space provided below.	For Examiner's Use
2.	Heat the tube and content <b>gently</b> for 5 minutes.	
Use hea	e a <b>luminous</b> Bunsen flame with the air-hole <b>closed</b> for this purpose. Ensure even ating of the sample and boiling tube.	
3.	Place the boiling tube into a <b>dry</b> 250 cm <sup>3</sup> beaker to cool.	
You	u may wish to proceed with other experiments while waiting for the boiling tube to cool.	
4.	Weigh and record the mass of the cooled boiling tube containing the residue.	
5.	Repeat points 2 (heat gently for 2 minutes subsequently) to 4 as necessary until a constant mass is obtained.	
6.	Turn off the Bunsen burner.	
(i)	In an appropriate format in the space below, record all weighings.	
	[4]	
	) Using your results, calculate the mass lost upon decomposition of <b>FA 4</b> .	
(11)	Osing your results, calculate the mass lost upon decomposition of PA 4.	
	mass lost =[1]	
(ii	i) Using your answer to 2(b)(ii), calculate the percentage mass lost.	

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percentage mass lost = .....[1]

<ul><li>(iv) Using your answer to 2(b)(iii), determine whether the decomposition reaction 4 or reaction 5</li></ul>	occurs <i>via</i>	For Examiner's Use
	<b>-</b>	
	••••••	
	[2]	
(v) Using your answer to 2(b)(ii) and 2(b)(iv), calculate the amount of CO	<sub>2</sub> lost.	
amount of CO <sub>2</sub> lost =	[1]	
(vi) Using your answer to 2(b)(v), calculate the percentage purity of NaHC	O <sub>3</sub> in <b>FA 4</b> .	
		:
percentage purity of NaHCO <sub>3</sub> in FA 4 =	[2]	

#### (c) Planning

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The percentage purity of NaHCO<sub>3</sub> in **FA 4** was determined *via* volatilisation gravimetric analysis in **2(b)**. Titrimetric analysis such as the thermometric titration in **1(a)** using **reaction 1** can also be used. In addition, the more usual acid-base titrations based on either **reaction 1** or **reaction 2** are applicable too.

reaction 1 NaHCO<sub>3</sub>(aq) + NaOH(aq) 
$$\rightarrow$$
 Na<sub>2</sub>CO<sub>3</sub>(aq) + H<sub>2</sub>O( $l$ )

reaction 2 NaHCO<sub>3</sub>(aq) + 
$$\frac{1}{2}$$
 H<sub>2</sub>SO<sub>4</sub>(aq)  $\rightarrow \frac{1}{2}$  Na<sub>2</sub>SO<sub>4</sub>(aq) + H<sub>2</sub>O( $l$ ) + CO<sub>2</sub>(g)

Plan an investigation to determine the percentage purity of NaHCO<sub>3</sub> in **FA 4** using an acid-base titration involving **reaction 2**.

You may assume that you are provided with:

- solid FA 4, impure NaHCO<sub>3</sub> of greater than 95% purity
- 0.200 mol dm<sup>-3</sup> sulfuric acid, H₂SO<sub>4</sub>
- acid-base indicator of your choice
- 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 indicator you would use, including the colour change at the endpoint,
- · the measurements you would make,
- an outline of how you would use your results to determine the percentage purity of NaHCO<sub>3</sub> in FA 4.

[A <sub>r</sub> : Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

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[8]	
[Total: 22]	

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## 3 Qualitative Analysis

FA 5 contains two unknown cations.

FA 6 contains one unknown anion.

(a) Perform the tests described in Table 3.1, and record your observations in the table. Test and identify any gases evolved. If there is no observable change, write no observable change.

Table 3.1

observations tests 1. To 2 cm depth of FA 5 in a test tube, add aqueous sodium hydroxide. Warm the mixture. Filter the mixture and use the resulting filtrate for Test 2 and residue for Test 3. To 1 cm depth of the filtrate from 2. Test 1, add dilute nitric acid dropwise with shaking until in excess. Add aqueous ammonia. Pour nitric acid onto the residue on the 3. filter paper from Test 1. Divide the liquid that filters through into 2 equal portions. To one portion, add aqueous sodium hydroxide slowly with shaking. To the other portion of the liquid from 4. Test 3, add aqueous ammonia. Place about 2 cm depth of the FA 6 5. solution in a test-tube. To this solution, add about 2 cm depth of FA 5.

[6]

For

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(b)	Deduce the unknown ions present in the following solutions.  For Examir.				
	(i)	FA 5	Use		
			1		
		[2]			
	(ii)	FA 6			
		[1]			
(c)	Wr	ite equations to account for the observations in Test 5.			
	•	[2]			
		[Total: 11]			
		(voidin vi)			

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# Qualitative Analysis Notes [ppt. = precipitate]

# (a) Reactions of aqueous cations

	reaction with	
cation	NaOH(aq)	NH₃(aq)
aluminium, A <i>l</i> ³⁺(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH <sub>4</sub> <sup>+</sup> (aq)	ammonia produced on heating	_
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca <sup>2+</sup> (aq)	white, ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu <sup>2+</sup> (aq),	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe²+(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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg²⁺(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn <sup>2+</sup> (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 <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess

# (b) Reactions of anions

nion reaction		
carbonate,	CO <sub>2</sub> liberated by dilute acids	
chloride, C <i>l</i> <sup>-</sup> (aq)	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in NH <sub>3</sub> (aq))	
bromide, Br <sup>-</sup> (aq)	gives pale cream ppt. with Ag <sup>+</sup> (aq) (partially soluble in NH <sub>3</sub> (aq))	
iodide, I <sup>-</sup> (aq)	gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq))	
nitrate, NO₃(aq)	NH <sub>3</sub> liberated on heating with OH <sup>-</sup> (aq) and A <sup>I</sup> foil	
nitrite, NO <sub>2</sub> (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 <sub>4</sub> <sup>2-</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (insoluble in excess dilute strong acids)	
sulfite, SO <sub>3</sub> <sup>2-</sup> (aq)	SO <sub>2</sub> liberated with dilute acids; gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in dilute strong acids)	

# (c) Tests for gases

gas	test and test result	
ammonia, NH <sub>3</sub>	turns damp red litmus paper blue	
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )	
chlorine, Cl <sub>2</sub>	bleaches damp litmus paper	
hydrogen, H <sub>2</sub>	"pops" with a lighted splint	
oxygen, O <sub>2</sub>	relights a glowing splint	
sulfur dioxide, SO <sub>2</sub>	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 <sub>2</sub>	greenish yellow gas	pale yellow	pale yellow
bromine, Br <sub>2</sub>	reddish brown gas / liquid	orange	orange-red
iodine, I <sub>2</sub>	black solid / purple gas	brown	purple



# EUNOIA JUNIOR COLLEGE JC2 Preliminary Examination 2021 General Certificate of Education Advanced Level Higher 2

CANDIDATE NAME		
CIVICS GROUP		INDEX NUMBER
CHEMISTE	RY	9729/04
Paper 4 Practica	al	31 August 2021
		2 hours 30 minutes
Candidates ansv	ver on the Question Paper.	
Additional Materi	ials: As listed in the Confidental Instructions	

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## Answer all the questions in the spaces provided.

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# 1 Investigation on the behaviour of acids and bases in aqueous solution

FA 1 is 1.00 mol dm<sup>-3</sup> sodium hydrogencarbonate, NaHCO<sub>3</sub>

FA 2 is sodium hydroxide, NaOH, of concentration between 1.5 mol dm<sup>-3</sup> and 2.0 mol dm<sup>-3</sup>

FA 3 is 2.00 mol dm<sup>-3</sup> sulfuric acid, H<sub>2</sub>SO<sub>4</sub>

According to the Arrhenius theory, an acid-base neutralisation reaction involves reacting together  $H^{+}(aq)$  and  $OH^{-}(aq)$  to produce water molecules.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

 $\Delta H_{\text{neutralisation}} = -57.1 \text{ kJ mol}^{-1}$ 

This task involves two different acid-base reactions.

The first reaction is between sodium hydrogencarbonate, FA 1, and sodium hydroxide, FA 2.

reaction 1 NaHCO<sub>3</sub>(aq) + NaOH(aq) 
$$\rightarrow$$
 Na<sub>2</sub>CO<sub>3</sub>(aq) + H<sub>2</sub>O( $l$ )

ΔH<sub>reaction 1</sub>

The molar enthalpy change for reaction 1,  $\Delta H_{\text{reaction 1}}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with NaOH.

Instead of using an indicator to determine the endpoint, you will perform a thermometric titration to determine the equivalence point of the reaction. The equivalence point is that point where  $H^{\dagger}(aq)$  from the acid and  $OH^{-}(aq)$  from the base are present in equal molar amounts.

The second reaction is between sodium hydrogencarbonate, FA 1, and sulphuric acid, FA 3.

reaction 2 NaHCO<sub>3</sub>(aq) + 
$$\frac{1}{2}$$
 H<sub>2</sub>SO<sub>4</sub>(aq)  $\rightarrow \frac{1}{2}$  Na<sub>2</sub>SO<sub>4</sub>(aq) + H<sub>2</sub>O( $l$ ) + CO<sub>2</sub>(g)  $\Delta H_{\text{reaction 2}}$ 

The molar enthalpy change for reaction 2,  $\Delta H_{\text{reaction 2}}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with H<sub>2</sub>SO<sub>4</sub>.

In your first experiment, you will perform a thermometric titration by adding portions of FA 2 progressively to a known volume of FA 1. You will continue adding FA 2 until the equivalence point is reacted and passed. Throughout the experiment you will note and record the temperature of the mixture after each addition.

You will then analyse your results graphically in order to determine the

- titration volume at the equivalence point, Vequivalence,
- maximum temperature change, ΔT<sub>maximum 1</sub>,
- molar enthalpy change, ΔH<sub>reaction 1</sub>, for reaction 1.

In your second experiment, you will mix together a given volume of **FA 1** with a suitable volume of **FA 3**.

You will then determine the

- maximum temperature change, ΔT<sub>maximum 2</sub>,
- molar enthalpy change, ΔH<sub>reaction 2</sub>, for reaction 2.

#### (a) The reaction between FA 1 and FA 2

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In this task you will need to record the maximum temperature of the reaction mixture when specified volumes of **FA 2** have been added. It is important that the volume of **FA 2** recorded is the total volume you have added up to that point when the temperature reading was made.

Note: If you overshoot on an addition, record the **actual** total volume of **FA 2** added up to that point.

In an appropriate format in the space provided on page 4, record all values of temperature, T, and each total volume of **FA 2** added.

- (i) 1. Fill a burette with FA 2.
  - 2. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
  - 3. Using a pipette, transfer 25.0 cm<sup>3</sup> of **FA 1** to the first Styrofoam cup.
  - 4. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature.
  - 5. From the burette, add 2.00 cm<sup>3</sup> of **FA 2** to the cup and stir the mixture thoroughly.
  - 6. Read and record the maximum temperature of the mixture, *T*, and the volume of **FA 2** added.
  - 7. Repeat points 5 and 6 until a total of 30.00 cm<sup>3</sup> of **FA 2** has been added. After each addition of **FA 2**, record the maximum temperature of the mixture and the total volume of **FA 2** added up to that point.

# Results

For Examiner's Use

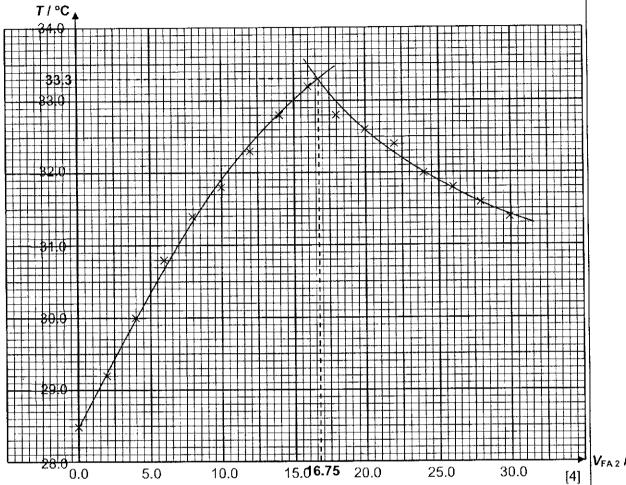
Total vol of FA2 added/ cm <sup>3</sup>	T/°C
0.00	28.5
2.00	29.2
4.00	30.0
6.00	30.8
8.00	31.4
10.00	31.8
12.00	32.3
14.00	32.8
16.00	33.2
18.00	32.8
20.00	32.6
22.00	32.4
24.00	32.0
26.00	31.8
28.00	31.6
30.00	31.4

[2]

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(ii) On the grid below, plot a graph of temperature, T (y-axis) against volume of FA 2 added (x-axis).

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V<sub>FA 2</sub> / cm<sup>3</sup>

- (iii) Draw two smooth lines of best fit.
  - The first best-fit line should be drawn using the plotted points where the temperature is rising.
  - The second best-fit line should be drawn using the plotted points where the temperature is falling.
  - Extrapolate these lines until they cross.

Note: Each line should have a shape best suited to its plotted points.

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		<ul> <li>the maximum temperature reached, T<sub>maximum</sub>,</li> <li>the maximum temperature change, ΔT<sub>maximum</sub> 1,</li> <li>the volume, V<sub>equivalence</sub>, of FA 2 needed to complete of FA 1.</li> </ul>	etely react with 25.0 cm³
		Show on your graph how you obtained these values	-
		Record these values in the spaces provided below.	
		maximum temperature reached, $T_{\text{maximum}} = \dots$	33.3 °C
		maximum temperature change, $\Delta T_{\text{maximum 1}} =$	4.8 °C
		volume of FA 2 used, V <sub>equivalence</sub> =	16.75 cm <sup>3</sup>
			[3]
(b)	lde	eally, the graph line <b>before</b> the equivalence point should be ncaves downward (that is, gradient of the curve decreases	e an <i>increasing</i> curve that ).
	(i)	Explain why the graph line is increasing.	
		As the reaction is exothermic, heat energy is released	for each addition, hence
		temperature increases with each addition.	
			[1]
	(ii)	Explain why the graph line concaves downward.	
		The same amount of heat energy is released for each	addition. However, <b>mass</b>
		of the mixture increases, hence causing a smaller te	mperature rise for each
		addition, leading to the graph line concaving downward	······································
			[1]

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# (c) The reaction between FA 1 and FA 3

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The molar enthalpy change for **reaction 2**,  $\Delta H_{reaction2}$ , is the enthalpy change when 1.00 mol of NaHCO<sub>3</sub> reacts completely with H<sub>2</sub>SO<sub>4</sub>.

In this task you will calculate a value for the molar enthalpy change,  $\Delta H_{\text{reaction 2}}$ . To do this, you will need to determine the maximum temperature change produced when measured volumes of **FA 1** and **FA 3** are mixed.

Upon considering the concentrations of **FA 1** and **FA 3**, 15 cm<sup>3</sup> of **FA 3** was chosen to add to 40 cm<sup>3</sup> of **FA 1**.

Note: You should be aware that the reaction mixture will produce considerable frothing.

(i)	Explain why 15 cm <sup>3</sup> of <b>FA 3</b> was chosen to add to 40 cm <sup>3</sup> of <b>FA 1</b> .
	There must be an excess of acid to completely react with the NaHCO <sub>3</sub> ,
	although too large a volume would diminish the temperature change caused by
	the reaction
	[1
(ii)	Identify the apparatus you intend to use to measure the volume of FA 1.
	Explain your choice.
	apparatus burette
	explanation NaHCO3 is the limiting reagent, so accurate/precise measurement
	of its volume is essential
	[1

(iii) In an appropriate format in the space provided below, record all values of measured temperature for this experiment.

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- 1. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
- Transfer 40 cm<sup>3</sup> of FA 1 into the first Styrofoam cup using the apparatus identified in 1(c)(ii).
- 3. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature,  $T_{\text{FA 1}}$ . This is the initial temperature of **FA 1**.
- 4. Wash and dry the thermometer.
- 5. Measure 15.0 cm $^3$  of **FA 3** using an appropriate measuring cylinder. Stir the **FA 3** solution in the measuring cylinder with the thermometer. Read and record its temperature,  $T_{\text{FA 3}}$ . This is the initial temperature of **FA 3**.
- 6. Carefully add the contents of the measuring cylinder to the Styrofoam cup in small portions to avoid too much frothing.
- 7. Place the lid on the cup and insert the thermometer through the lid. Stir the mixture.
- 8. Continue to stir the mixture. Measure and record the temperature,  $T_{\rm mixture}$  that shows the maximum change from the initial temperature.

#### Results

T <sub>FA 1</sub> /°C	28.8
T <sub>FA 3</sub> /°C	28.8
T <sub>mixture</sub> / °C	28.4

[1]

#### **Calculations**

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(d) For the purpose of these calculations, you should assume that the reaction mixture has a density of 1.00 g cm<sup>-3</sup> and a specific heat capacity, c, of 4.18 J g<sup>-1</sup> K<sup>-1</sup>.

Use your results from (a)(iv) to calculate

(i) the concentration of sodium hydroxide, [NaOH], in FA 2,

[NaOH] = 
$$\frac{25.0 \times 10^{-3}}{16.75 \times 10^{-3}} \times 1.00 = 1.493 \approx 1.49 \text{ mol dm}^{-3} \text{ (3 s.f.)}$$

[NaOH] in **FA 2** = 
$$\frac{1.49 \text{ mol dm}^{-3}}{1.49 \text{ mol dm}^{-3}}$$

(ii) the heat change, q, for the reaction occurring in (a)(i), and hence determine a value for the molar enthalpy change for reaction 1,  $\Delta H_{\text{reaction 1}}$ .

$$q = mc\Delta T_{\text{maximum 1}} = (25.0 + 16.75) \times 1.00 \times 4.18 \times 4.8 = 837.672 \approx 838 \text{ J } (3 \text{ s.f.})$$

$$n(\text{NaHCO}_3) = 25.0 \times 10^{-3} \text{ mol}$$
  

$$\Delta H_{\text{reaction 1}} = -\frac{837.672}{25.0 \times 10^{-3}} = -33506.88 \text{ J mol}^{-1} \approx -33.5 \text{ kJ mol}^{-1} \text{ (3 s.f.)}$$

$$\Delta H_{\text{reaction 1}} = \frac{-33.5 \text{ kJ mol}^{-1}}{[2]}$$

(e) Use your results from (c)(iii) to calculate a value for the molar enthalpy change for reaction 2, ΔH<sub>reaction 2</sub>. For the experiment in (c)(iii), the weighted average initial temperature, T<sub>average</sub>, of FA 1 and FA 3 may be calculated using the formula given below.

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$$T_{\text{average}} = \frac{(V_{\text{FA1}} \times T_{\text{FA1}}) + (V_{\text{FA3}} \times T_{\text{FA3}})}{(V_{\text{FA1}} + V_{\text{FA3}})}$$
$$T_{\text{average}} = \frac{(40 \times 28.8) + (15 \times 28.8)}{40 + 15} = 28.8 \, ^{\circ}\text{C}$$

$$\Delta T_{\text{maximum 2}} = |28.8 - 28.4| = 0.4 \text{ °C}$$

$$q = mc\Delta T_{\text{maximum 2}} = (40.0 + 15.0) \times 1.00 \times 4.18 \times 0.4 = 92.0 \text{ J}$$

$$n(\text{NaHCO}_3) = 40.0 \times 10^{-3} \text{ mol}$$
  

$$\Delta H_{\text{reaction 2}} = +\frac{92.0}{40.0 \times 10^{-3}} = +2300 \text{ J mol}^{-1} = +2.30 \text{ kJ mol}^{-1} \text{ (3 s.f.)}$$

$$\Delta H_{\text{reaction 2}} = +2.30 \text{ kJ mol}^{-1}$$
 [1]

(f) Ionic equations for neutralisation, reaction 1, and reaction 2 are shown below.

$$H^+ + OH^- \rightarrow H_2O$$
  $\Delta H_{\text{neutralisation}} = -57.1 \text{ kJ mol}^{-1}$ 
 $HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$   $\Delta H_{\text{reaction 1}}$ 
 $HCO_3^- + H^+ \rightarrow H_2O + CO_2$   $\Delta H_{\text{reaction 2}}$ 

Carbon dioxide may be removed from stale air by bubbling the air through aqueous alkali. An equation for this reaction is given below.

reaction 3 
$$2OH^{-}(aq) + CO_{2}(g) \rightarrow CO_{3}^{2-}(aq) + H_{2}O(l)$$
  $\Delta H_{reaction 3}$ 

Use your answers to **(d)(ii)** and **(e)**, together with the molar enthalpy change of neutralisation,  $\Delta H_{\text{neutralisation}}$ , to determine a value for the molar enthalpy change for this reaction,  $\Delta H_{\text{reaction 3-}}$ 

$$\begin{array}{ll} HCO_{3}^{-}(aq) + OH^{-}(aq) \rightarrow CO_{3}^{2-}(aq) + H_{2}O(\textit{1}) & \Delta H_{\text{reaction 1}} \\ H_{2}O(\textit{1}) + CO_{2}(g) \rightarrow HCO_{3}^{-}(aq) + H^{+}(aq) & -\Delta H_{\text{reaction 2}} \\ H^{+}(aq) + OH^{-}(aq) \rightarrow H_{2}O(\textit{1}) & -57.1 \\ \hline \\ 2OH^{-}(aq) + CO_{2}(g) \rightarrow CO_{3}^{2-}(aq) + H_{2}O(\textit{1}) & \Delta H_{\text{reaction 3}} \end{array}$$

$$\Delta H_{\text{reaction 3}} = \Delta H_{\text{reaction 1}} - \Delta H_{\text{reaction 2}} - 57.1 = -33.5 - (+2.09) -57.1$$
  
= -92.69 \approx -92.7 kJ mol<sup>-1</sup>

$$\Delta H_{\text{reaction 3}} = -92.7 \text{ kJ mol}^{-1}$$
 [1]

(g) Another student planned a different thermometric titration experiment to determine the value of the titration volume at the equivalence point,  $V_{\text{equivalence}}$ , and the maximum temperature change,  $\Delta T_{\text{maximum 1}}$ , for **reaction 1**.

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In this other experiment, different volumes of **FA 1** and **FA 2** as shown in Table 1.1 are mixed, and the maximum temperature change,  $\Delta T_{\text{max}}$ , is determined for each mixture.

Table 1.1

experiment	1	2	3	4	5	6	7	8
V <sub>FA 2</sub> / cm <sup>3</sup>	10.00	15.00	20.00	25.00	30.00	35.00	40.00	45.00
V <sub>FA 1</sub> / cm <sup>3</sup>	45.00	40.00	35.00	30.00	25.00	20.00	15.00	10.00

 $\Delta T_{\text{max}}$  is plotted against the volume of **FA 2** used,  $V_{\text{FA 2}}$ .  $V_{\text{equivalence}}$  and  $\Delta T_{\text{maximum 1}}$  are determined from the intersection of the two straight lines drawn as shown in Fig. 1.1.

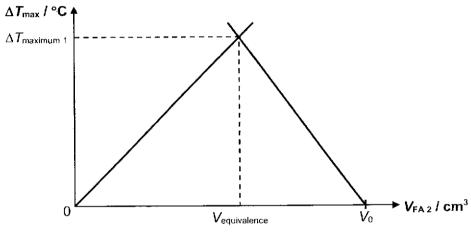


Fig. 1.1

(i) Suggest a value for  $V_0$  in Fig. 1.1.

$$V_0 =$$
 55.0 cm<sup>3</sup> [1]

(ii) The student claimed that this method gives a more accurate value of  $V_{\text{equivalence}}$  and  $\Delta T_{\text{maximum 1}}$  compared to the method in 1(a).

Do you agree with the student? Explain.

V<sub>equivalence</sub> Agree. This is because there is greater uncertainty in the extrapolation of a curve than of a straight line. *OR* 

Disagree. This is because there are more data points in 1(a), allowing for better extrapolation of the line / Both methods uses the same graphical extrapolation treatment to determine  $V_{\text{advivalence}}$ .

	For
AT <sub>maximum 1</sub> Agree. This is because there is greater heat lost to the	Examinei Use
surroundings for the method in 1(a) as FA 2 is added to FA 1 in fifteen 2 cm <sup>3</sup>	
portions over a prolonged period of time.	
[2]	:
[Total: 22]	

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2 Determination of identity of the impurity in FA 4, the decomposition route of NaHCO<sub>3</sub>, the percentage purity of FA 4

For Examiner's

**Solid FA 4** is anhydrous sodium hydrogencarbonate, NaHCO<sub>3</sub>, of greater than 95% purity that has been contaminated with a sodium halide.

You are also provided with an aqueous solution of FA 4, labelled FA 4 solution.

- (a) You will devise and perform a simple test, based on the Qualitative Analysis Notes on pages 23–24, to identify the halide present in the FA 4 solution.
  - (i) Describe a test, using only the bench reagents provided, which will allow you to identify the halide impurity present in **FA 4**.

Add AgNO<sub>3</sub>(ag) dropwise to 1 cm<sup>3</sup> of FA 4 solution. After the ppt is formed, add aqueous ammonia. [1]

(ii) Perform the test you described in 2(a)(i) using the FA 4 solution provided.

Record your observations and hence deduce the identity of the halide in **solid FA 4**.

Upon addition of AgNO<sub>3</sub>(aq) dropwise, a white ppt is formed which is soluble in excess aqueous ammonia.

identity of halide chloride [2]

## (b) Thermal decomposition of NaHCO<sub>3</sub>

When NaHCO<sub>3</sub> is heated above 110  $^{\circ}$ C (but not heated to "red heat") it has been observed that both H<sub>2</sub>O(g) and CO<sub>2</sub>(g) are evolved, and that after this decomposition is complete, a white solid residue remains. Among possible reactions, the two following reactions seem the ones that are most likely to explain these facts:

reaction 4  $2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$ 

reaction 5  $2NaHCO_3(s) \rightarrow Na_2O(s) + H_2O(g) + 2CO_2(g)$ 

Sodium halides are stable to heat.

In this experiment, solid **FA 4** is heated gently in a boiling tube, over a luminous Bunsen flame, until **all** NaHCO<sub>3</sub> has been decomposed. The data will be used to determine

- the percentage mass loss,
- whether the decomposition takes place via reaction 4 or reaction 5,
- the amount of CO<sub>2</sub> lost,
- the percentage purity of NaHCO<sub>3</sub> in FA 4

[Ar: Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

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1. Weigh accurately between 2.00 g and 2.50 g of **FA 4** in the boiling tube provided. Record your weighings in the space provided below.

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2. Heat the tube and content **gently** for 5 minutes.

Use a **luminous** Bunsen flame with the air-hole **closed** for this purpose. Ensure even heating of the sample and boiling tube.

3. Place the boiling tube into a dry 250 cm³ beaker to cool.

You may wish to proceed with other experiments while waiting for the boiling tube to cool.

- 4. Weigh and record the mass of the cooled boiling tube containing the residue.
- 5. Repeat points 2 (heat gently for 2 minutes subsequently) to 4 as necessary until a constant mass is obtained.
- 6. Turn off the Bunsen burner.
- (i) In an appropriate format in the space below, record all weighings.

mass of empty boiling tube / g	30.000
mass of boiling tube + FA 4/g	32.290
mass of FA 4/g	2.290
mass of boiling tube + residue after first heating / g	31.534
mass of boiling tube + residue after second heating / g	31.530

[4]

(ii) Using your results, calculate the mass lost upon decomposition of FA 4.

Mass lost due to decomposition = 2.290 - 1.530 = 0.760 g

mass lost = 
$$0.760 \text{ g}$$
 [1]

(iii) Using your answer to 2(b)(ii), calculate the percentage mass lost.

% mass lost = 
$$0.760 / 2.290 \times 100\% = 33.19 \approx 33.2 \% (3 s.f.)$$

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(iv)	Using your answer to <b>2(b)(iii)</b> , determine whether the decomposition occurs <i>via</i> reaction <b>4</b> or reaction <b>5</b>	For Examiner's Use
	via reaction 4 :	
	% mass lost = (18+44) ÷ (2×84) × 100% = 36.9%	
	via reaction 5 :	
	% mass lost = (18 + 2×44) ÷ (2×84) × 100% = 63.1%	
	Since % mass lost is 33.2%, the decomposition occurs via reaction 4.	
	[2]	į.
(v)	Using your answer to 2(b)(ii) and 2(b)(iv), calculate the amount of CO <sub>2</sub> lost.	
	$n(CO_2)$ lost = 0.760 ÷ (18+44) = 0.012258 $\approx$ 0.0123 mol	
		5
	amount of $CO_2$ lost = $0.0123$ mol [1]	
(vi)	Using your answer to <b>2(b)(v)</b> , calculate the percentage purity of NaHCO₃ in <b>FA 4</b> .	
	$n(NaHCO_3) = 2n(CO_2) = 0.024516 \text{ mol}$ mass of NaHCO <sub>3</sub> = $n(NaHCO_3) \times 84.0 = 2.0594 \text{ g}$ % purity = $2.0594 \div 2.290 \times 100\% = 89.9\%$	
	porcentage purity of NaHCO <sub>3</sub> in <b>FA 4</b> =	

#### (c) Planning

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The percentage purity of NaHCO<sub>3</sub> in FA 4 was determined *via* volatilisation gravimetric analysis in 2(b). Titrimetric analysis such as the thermometric titration in 1(a) using reaction 1 can also be used. In addition, the more usual acid-base titrations based on either reaction 1 or reaction 2 are applicable too.

reaction 1 NaHCO<sub>3</sub>(aq) + NaOH(aq) 
$$\rightarrow$$
 Na<sub>2</sub>CO<sub>3</sub>(aq) + H<sub>2</sub>O( $l$ )

reaction 2 NaHCO<sub>3</sub>(aq) + 
$$\frac{1}{2}$$
 H<sub>2</sub>SO<sub>4</sub>(aq)  $\rightarrow \frac{1}{2}$  Na<sub>2</sub>SO<sub>4</sub>(aq) + H<sub>2</sub>O(*l*) + CO<sub>2</sub>(g)

Plan an investigation to determine the percentage purity of NaHCO<sub>3</sub> in **FA 4** using an acid-base titration involving **reaction 2**.

You may assume that you are provided with:

- solid FA 4, impure NaHCO<sub>3</sub> of greater than 95% purity
- 0.200 mol dm<sup>-3</sup> sulfuric acid, H<sub>2</sub>SO<sub>4</sub>
- acid-base indicator of your choice
- 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 indicator you would use, including the colour change at the endpoint,
- the measurements you would make,
- an outline of how you would use your results to determine the percentage purity of NaHCO<sub>3</sub> in **FA 4**.

[A <sub>r</sub> : Na, 23.0; O, 16.0; C, 12.0; H, 1.0]
Pre-calculation:
Using a 50 cm³ burette, a 25 cm³ pipette, and a 250 cm³ volumetric flask
Assuming that a titre value of 25.00 cm <sup>3</sup> of 0.200 mol dm <sup>-3</sup> H <sub>2</sub> SO <sub>4</sub> .
$n(H_2SO_4)$ in 25.00 cm <sup>3</sup> = 25.00 × 10 <sup>-3</sup> × 0.200 = 0.00500 mol
$n(NaHCO_3)$ in 25.0 cm <sup>3</sup> aliquot = $2n(H_2SO_4) = 0.0100$ mol
n(NaHCO <sub>3</sub> ) in 250 cm <sup>3</sup> solution prepared = 0.100 mol
$m(NaHCO_3)$ present = $0.100 \times (23.0+1.0+12.0+16.0\times3) = 8.40 g$
maximum mass of <b>FA 4</b> measured = 8.40 ÷ 0.95 = <b>8.84 g</b>

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Proc	edure:
1	Weigh out accurately about 8.84 g of FA 4 into a clean, dry weighing bottle
<b>.</b>	using an electronic balance.
2	Transfer all of the solid into a 250 cm <sup>3</sup> beaker and dissolve it in about 100 cm <sup>3</sup>
	of deionised water.
3	Transfer the solution to a 250 cm <sup>3</sup> volumetric flask, labelled FA 4 solution.
	Rinse the beaker with deionised water several times, adding each rinsing to the
	volumetric flask.
4	Fill a 50 cm³ burette with 0.200 mol dm⁻³ H₂SO₄.
5	Use a pipette to transfer 25.0 cm <sup>3</sup> of FA 4 solution into a 250 cm <sup>3</sup> conical flask.
6	Add three drops of methyl orange indicator into the conical flask.
7	Titrate the FA 4 solution in the flask with 0.200 mol dm <sup>-3</sup> sulfuric acid. The
	endpoint is reached when the solution changes from yellow to orange.
8	Record the titration results in a table.
	Final burette reading / cm <sup>3</sup>
	Initial burette reading / cm³
	Volume of 0.200 mol dm <sup>-3</sup> H <sub>2</sub> SO <sub>4</sub> added / cm <sup>3</sup>
9.	Repeat points 5 to 8 until consistent titre values within ±0.10 cm³ are obtained.
•••••	
******	
•••••	

Calculation:
1. From the consistent titration results, calculate the average volume of 0.200 mol
dm <sup>-3</sup> H <sub>2</sub> SO <sub>4</sub> , V cm <sup>3</sup> .
2. $n(H_2SO_4)$ used = $V \times 10^{-3} \times 0.200 = 2V \times 10^{-4}$ mol
3. $n(NaHCO_3)$ present in 25.0 cm <sup>3</sup> = $2n(H_2SO_4) = 4V \times 10^{-4}$ mol
4. $n(NaHCO_3)$ present in 250 cm <sup>3</sup> = 4 $V \times 10^{-3}$ mol
5. $m(NaHCO_3)$ present in sample weighed = $n(NaHCO_3) \times 84.0 = 0.336V$ g
6. Assuming m g of FA 4 weighed out,
Percentage purity = 0.336V ÷ m × 100% = 33.6V/m %
······································
······································
[8]
[Total: 22]

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### 3 Qualitative Analysis

For Examiner's Use

- FA 5 contains two unknown cations.
- FA 6 contains one unknown anion.
- (a) Perform the tests described in Table 3.1, and record your observations in the table. Test and identify any gases evolved. If there is no observable change, write no observable change.

Table 3.1

	tests	observations
1.	To 2 cm depth of <b>FA 5</b> in a test tube, add aqueous sodium hydroxide.	White ppt, soluble in excess NaOH Reddish-brown ppt, insoluble in excess NaOH
	Warm the mixture.	Damp red litmus paper remained red.
	Filter the mixture and use the resulting filtrate for <b>Test 2</b> and residue for <b>Test 3</b> .	Colourless filtrate with reddish-brown residue.
2.	To 1 cm depth of the filtrate from Test 1, add dilute nitric acid dropwise with shaking until in excess.	White ppt, soluble in excess HNO <sub>3</sub> to give a colourless solution.
	Add aqueous ammonia.	White ppt insoluble in excess NH <sub>3</sub>
3.	Pour nitric acid onto the residue on the filter paper from <b>Test 1</b> . Divide the liquid that filters through into 2 equal portions.	Reddish-brown ppt dissolves to give a yellow solution.
	To one portion, add aqueous sodium hydroxide slowly with shaking.	Reddish-brown ppt, insoluble in excess NaOl-
4.	To the other portion of the liquid from Test 3, add aqueous ammonia.	Reddish-brown ppt, insoluble in excess NH <sub>3</sub>
5.	Place about 2 cm depth of the <b>FA 6</b> solution in a test-tube.	
	To this solution, add about 2 cm depth of FA 5.	Pale brown/ beige ppt Effervescence of colourless gas that formed white ppt with Ca(OH) <sub>2</sub>

[6]

(b)	(b) Deduce the unknown ions present in the following solutions.		
	(i)	FA 5	
		The unknown ions are $Al^{3+}$ and $Fe^{3+}$ .	
		When NaOH is added, Al3+ forms white ppt which is soluble in excess NaOH.	
		When aqueous ammonia is added, $Al^{\beta^+}$ forms white ppt which is insoluble in	
		excess ammonia	
		When NaOH and ammonia are added separately to Fe3+, a reddish brown ppt.	
		is formed and is insoluble in excess base.	
		[2]	
	(ii)	FA 6	
		The unknown ion is CO <sub>3</sub> <sup>2-</sup> . Accept HCO <sub>3</sub> <sup>-</sup> .	
		When the acidic FA 5 solution is added, carbon dioxide is released.	
		[1]	
(c)	Wr	ite equations to account for the observations in Test 5.	
	[A	$l(H_2O)_6]^{3+} + H_2O \rightleftharpoons [Al(H_2O)_5(OH)]^{2+} + H_3O^+ \text{ or}$	
	[F	$e(H_2O)_6]^{3+} + H_2O \rightleftharpoons [Fe(H_2O)_5(OH)]^{2+} + H_3O^+$	
	••••		
	21	1"+ ℃O <sub>3</sub> 2····································	
		[2]	
		[Total: 11]	

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# **Qualitative Analysis Notes** [ppt. = precipitate]

### (a) Reactions of aqueous cations

	reaction with		
cation	NaOH(aq)	NH₃(aq)	
aluminium, $A\hat{\ell}^{+}(aq)$	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH <sub>4</sub> <sup>†</sup> (aq)	ammonia produced on heating	_	
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.	
calcium, Ca <sup>2+</sup> (aq)	white. ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.	
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess	
copper(II), Cu <sup>2+</sup> (aq),	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution	
iron(II), Fe <sup>2+</sup> (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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess	
magnesium, Mg²⁺(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, Zn <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess	

## (b) Reactions of anions

anion	reaction	
carbonate,	CO <sub>2</sub> liberated by dilute acids	
chloride, C <i>l</i> <sup>-</sup> (aq)	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in NH <sub>3</sub> (aq))	
bromide, Br <sup>-</sup> (aq)	gives pale cream ppt. with Ag⁺(aq) (partially soluble in NH₃(aq))	
iodide, I <sup>-</sup> (aq)	gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq))	
ni <b>tra</b> te, NO <sub>3</sub> (aq)	NH <sub>3</sub> liberated on heating with OH <sup>-</sup> (aq) and A <i>l</i> foil	
nitrite, NO <sub>2</sub> (aq)	$NH_3$ liberated on heating with $OH^-(aq)$ and $AI$ foil; NO liberated by dilute acids (colourless $NO \rightarrow$ (pale) brown $NO_2$ in air)	
sulfate, gives white ppt. with Ba <sup>2+</sup> (aq) (insoluble in excess dilute strong a SO <sub>4</sub> <sup>2-</sup> (aq)		
sulfite, $SO_2$ liberated 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 <sub>3</sub>	turns damp red litmus paper blue	
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )	
chlorine, Cl <sub>2</sub>	bleaches damp litmus paper	
hydrogen, H₂	"pops" with a lighted splint	
oxygen, O <sub>2</sub>	relights a glowing splint	
sulfur dioxide, SO <sub>2</sub> turns aqueous acidified potassium manganate(VII) from purple to co		

### (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 <sub>2</sub>	reddish brown gas / liquid	orange	orange-red
iodine, I <sub>2</sub>	black solid / purple gas	brown	purple