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

1 Investigation of an organic compound and an inorganic compound

FA 1 is an aqueous solution of an organic compound, **Y**, which contains two different functional groups.

FA 2 contains two cations and one anion.

You will perform tests to identify:
- the functional groups in **Y** and hence deduce its possible structure
- the ions present in **FA 2**.

(a) (i) Carry out the following tests. Carefully record your observations in Table 1.1.
The volumes given below are approximate and should be estimated rather than measured.

Test and identify any gases evolved.

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

Table 1.1

	tests	observations
1	To a 1 cm depth of dilute sulfuric acid in a test-tube, add 2 cm of aqueous potassium manganate(VII). Then add 1 cm depth of **FA 1**. Allow to stand for 10 minutes, with occasional shaking. Continue with the remaining parts of Question 1.	Upon addition of FA1, purple manganate (VII) decolourises On standing, solution remains colourless. Some effervescence observed
2	To a 2 cm depth of **FA 1** in a test-tube, add all of the solid sodium carbonate provided in the stoppered tube.	Effervescence of gas that gives white ppt with limewater. The gas is CO_2
3	Test solution **FA 1** with Universal Indicator paper.	Universal indicator paper turns dark red. The colour corresponds to pH 1. 

[3]

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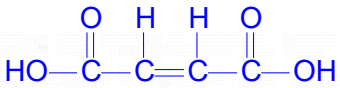
(ii) The molecular formula of **Y** is $C_4H_4O_4$. Draw one possible structure for **Y**. Use your observations in Table 1.1.

[Note: reaction with acidified $KMnO_4$ at room temperature: mild oxidation of alkene C=C

Reaction with Na_2CO_3 and pH1: acid-carbonate reaction, presence of carboxylic acid −COOH]



[1]

(b) (i) To a 1 cm depth of **FA 2** in a test-tube, add aqueous ammonia slowly, with shaking, until no further change is seen. Record all your observations.

Green ppt formed is insoluble in excess $NH_3(aq)$

The green ppt turns brown upon standing.

[2]

(ii) Deduce the identity of one of the cations in **FA 2** using your results in **(b)(i)**.

Fe^{2+}

[1]

[Note: green ppt is $Fe(OH)_2$, which is oxidized by air to brown ppt $Fe(OH)_3$]

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(iii) Devise and perform a series of simple tests to identify the anion and the remaining cation in **FA 2.** Your tests should be based on the Qualitative Analysis Notes on pages 15–16 and should use only the bench reagents provided. Record your tests and observations in the space below.

Any test requiring heating MUST be performed in a boiling tube.

The remaining cation does not form an insoluble hydroxide in excess sodium hydroxide. The anion is not a nitrate, nitrite or sulfite ion.

To test for cation:

To 1cm depth of FA2, add NaOH(aq) in excess. Filter the suspension and collect the filtrate for subsequent tests. [to isolate the 2nd cation in the filtrate]	Green residue that turns brown on standing. Filtrate is colourless.
To test for Ba^{2+}/Ca^{2+}: To 1cm depth of filtrate, add excess H_2SO_4 (aq) [to check for ppt of $BaSO_4$ or $CaSO_4$]	No ppt observed.
To test for Al^{3+}/Zn^{2+}: To 1cm depth of filtrate, add HNO_3 (aq) dropwise until no further change [to check for ppt of $Al(OH)_3$ or $Zn(OH)_2$]	No ppt observed. [If $[Al(OH)_4]^-$ or $[Zn(OH)_4]^{2-}$ is in the filtrate, slow addition of H^+ will remove the excess OH^- from the complex ion and produce white ppt of $Al(OH)_3$ or $Zn(OH)_2$]
To test for NH_4^+: Add 1cm depth of filtrate into a boiling tube, warm gently using the Bunsen burner [the filtrate already contains NaOH]	Gas produced turned moist litmus paper blue. The gas is NH_3 [$NH_4^+ + OH^- \longrightarrow NH_3 + H_2O$]

To test for anion:

To test for CO_3^{2-}: To 1cm depth of FA2, add HNO_3(aq)	No effervescence observed.
To test for SO_4^{2-}: To 1cm depth of FA2, add $Ba(NO_3)_2$(aq), followed by excess HNO_3(aq)	White ppt formed, insoluble in excess HNO_3. [to confirm SO_4^{2-}, we need to check that the ppt is insoluble in strong acid]
To test for Cl^-, Br^-, I^-: To 1cm depth of FA2, add $AgNO_3$(aq), followed by excess NH_3(aq)	No white/yellow ppt observed. [Ag^+ reacted with Fe^{2+} in FA2 to give a grey ppt via redox reaction $Ag^+ + Fe^{2+} \longrightarrow Ag + Fe^{3+}$]

[5]

(iv) Use your observations in **(b)(iii)** to deduce the identity of the other cation and the anion in **FA 2.**

cation NH_4^+ .....

anion SO_4^{2-} .....

[2]

[Total: 14]

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2 Determination of the molar enthalpy change of a reaction by an indirect method

Sodium hydrogencarbonate decomposes according to equation 1.

equation 1 $2NaHCO_3 \rightarrow Na_2CO_3 + CO_2 + H_2O$ ΔH_1

Sodium carbonate and sodium hydrogencarbonate each react with hydrochloric acid.

equation 2 $Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$ ΔH_2

equation 3 $NaHCO_3 + HCl \rightarrow NaCl + CO_2 + H_2O$ ΔH_3

In this question, you will perform an experiment to determine a value for ΔH_2. You will use data provided to calculate ΔH_3 and hence a value for ΔH_1.

You are provided with:
- solid **FA 3**, Na_2CO_3
- **FA 4**, 2.0 mol dm^{-3} hydrochloric acid, HCl.

(a) (i) Determination of the molar enthalpy change of reaction, ΔH_2

In this experiment, you will measure the temperature of hydrochloric acid in a polystyrene cup at regular time intervals, before and after sodium carbonate is added. You will analyse your results graphically to obtain an accurate value for the temperature change caused by the reaction.

You will use this value to calculate the heat change, q, for the experiment and hence determine a value for the molar enthalpy change of the reaction, ΔH_2.

In the space provided on page 6, 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 the capped container containing solid **FA 3**. Record the mass in your table on page 6.

2. Place one polystyrene cup inside another polystyrene cup and place both in a glass beaker.

3. Use the measuring cylinder to transfer 50 cm^3 of **FA 4** (an excess) into the polystyrene cup.

4. Carefully stir the **FA 4** in the polystyrene cup with the thermometer. Read and record the temperature, T. Start the stopwatch (t = 0.0 min). The stopwatch must be left to run for the rest of the experiment.

5. Continue to stir **FA 4**. Read and record T every 0.5 minute for two minutes.

6. At **exactly** 2.5 minutes, transfer all the solid **FA 3** to the polystyrene cup. Stir the mixture but do not read T.

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7. Continue to stir the mixture. Read and record T at t = 3.0 min and every 0.5 min until t = 8.0 min.

8. Reweigh the empty capped container. Record this mass in your table below.

Results

Mass of **FA3** in capped container / g	9.429
Mass of empty capped container / g	5.272
Mass of **FA3** used / g	4.157

Time/min	Temp/ °C
0.0	30.2
0.5	30.2
1.0	30.2
1.5	30.2
2.0	30.2
2.5	-
3.0	36.0
3.5	36.2
4.0	36.1

Time/min	Temp/ °C
4.5	35.9
5.0	35.8
5.5	35.7
6.0	35.6
6.5	35.6
7.0	35.4
7.5	35.2
8.0	35.2

[3]

(ii) Plot a graph of temperature, T, on the y-axis, against time, t, on the x-axis, on the grid in Fig. 2.1.

Draw a best-fit straight line taking into account all of the points before t = 2.5 min.

Draw another best-fit straight line taking into account all of the points after the temperature of the mixture has started to fall steadily.

Extrapolate (extend) both lines to t = 2.5 min. [3]

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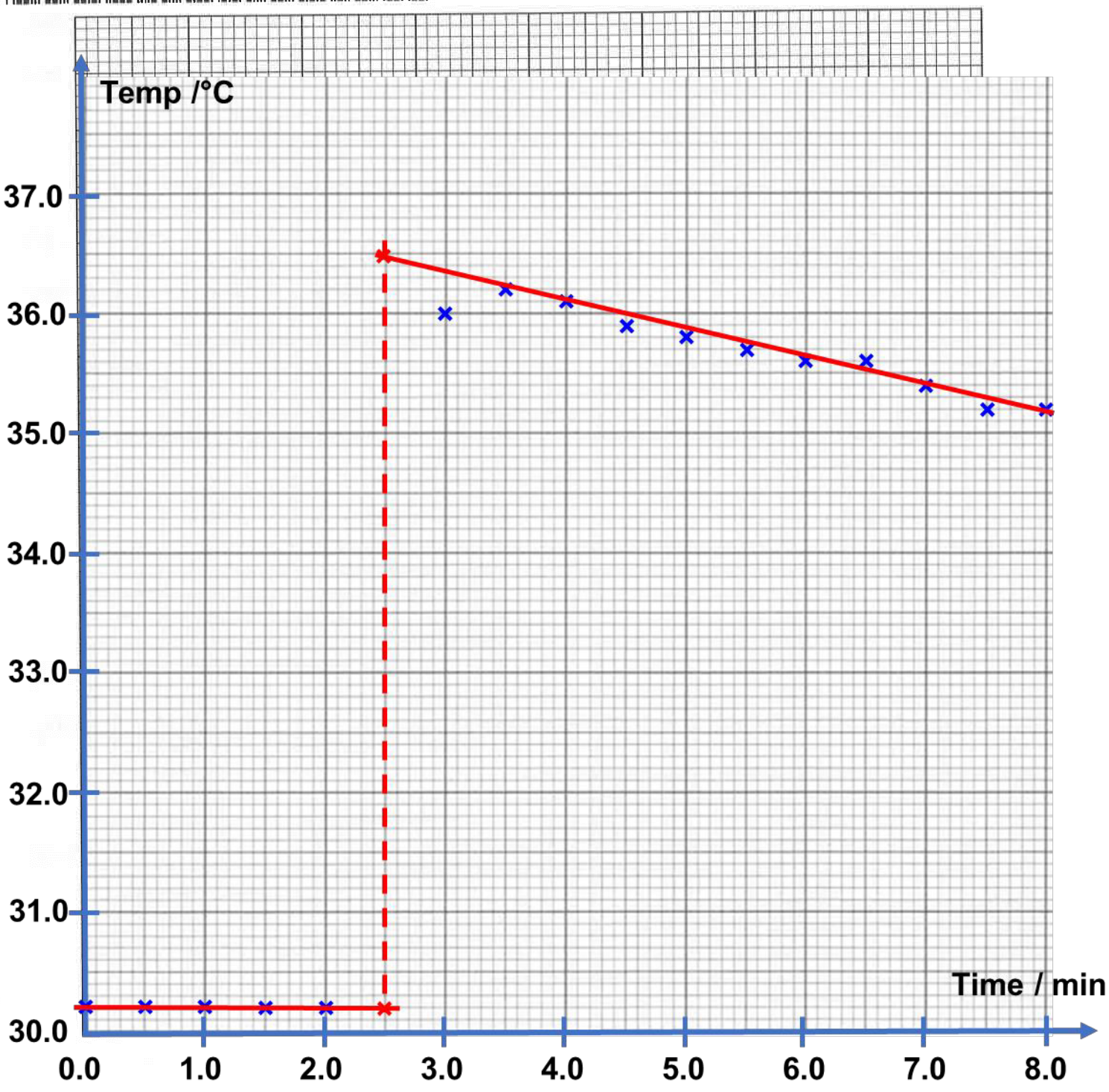

Fig. 2.1

(iii) From your graph, read the minimum temperature, T_{min}, and the maximum temperature, T_{max}, at t = 2.5 min. Record these values in the spaces below.

Calculate the temperature change, ΔT, at t = 2.5 min.

T_{min} = 30.2 °C .....

T_{max} = 36.5 °C .....

ΔT = +6.3 °C .....

[1]

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(iv) Calculate the heat change, q, for your experiment using the value you calculated in **(a)(iii)**.

Assume that the specific heat capacity of the reaction mixture is $4.18\,J\,g^{-1}\,K^{-1}$, and that the density of the reaction mixture is $1.00\,g\,cm^{-3}$.

Note: m is mass of aq solution ONLY.
50 cm^3 aq solution = 50 g

$q = m_{aq}c\Delta T$
$= 50 \times 4.18 \times (+6.3)$
$= 1316.7$ J
$= 1320$ J (3s.f.)

q = ... [1]

(v) Determine the molar enthalpy change, ΔH_2, for the reaction in equation 2. The hydrochloric acid is in excess.

Include the sign of ΔH_2 in your answer.

[A_r: C, 12.0 O, 16.0 Na, 23.0]

Amount of $Na_2CO_3 = \frac{4.157}{23.0 \times 2 + 12.0 + 16.0 \times 3} = 0.03922$ mol

$\Delta H_2 = -\frac{q}{\text{mol of limiting reagent}}$
$= -\frac{1316.7}{0.03922}$
$= -33572\ J\ mol^{-1} \approx -33.6\ kJ\ mol^{-1}$

ΔT is +ve, ΔH is -ve

ΔH_2 = ... [3]

(b) A student repeats the experiment, but uses 4.00 g of sodium hydrogencarbonate, $NaHCO_3$, instead of sodium carbonate.

The temperature **decreases** by 6.6 °C.

Determine the molar enthalpy change, ΔH_3, for the reaction in equation 3. The hydrochloric acid is in excess.

Include the sign of ΔH_3 in your answer.
[A_r: H, 1.0 C, 12.0 O, 16.0 Na, 23.0]

Amount of $NaHCO_3 = \frac{4.00}{23.0 + 1.0 + 12.0 + 16.0 \times 3} = 0.04762$ mol

$\Delta H_3 = -\frac{m_{aq}c\Delta T}{\text{mol of limiting reagent}}$
$= -\frac{50 \times 4.18 \times (-6.6)}{0.04762}$
$= +28967\ J\ mol^{-1} \approx +29.0\ kJ\ mol^{-1}$

ΔT is –ve, ΔH is +ve

ΔH_3 = ... [2]

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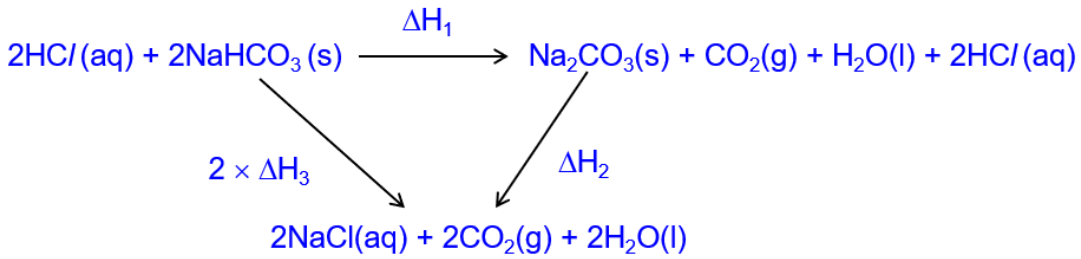
(c) Calculate a value for ΔH_1 for the reaction in equation 1.

Use your answers from **(a)(v)** and **(b)**.



By Hess's Law,

$\Delta H_1 = 2 \times \Delta H_3 - \Delta H_2$

$= 2 \times (+29.0) - (-33.6) = +91.6 \text{ kJ mol}^{-1}$

ΔH_1 = ... [5]

(d) Which piece of apparatus causes the greatest percentage error in your experiment? Explain why.

% error when measuring 50 cm^3 of FA4 using measuring cylinder = $\pm \frac{0.5}{50} \times 100 = \pm 1.0\%$

.....

% error when measuring ΔT using 0.2 °C thermometer = $\pm \frac{2 \times 0.1}{6.3} \times 100 = \pm 3.17\%$ [1]

(2 readings with 0.1 uncertainty to obtain ΔT)

[Total: 19]

Hence, the thermometer caused the greatest percentage error.

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3 Determination of the amount of water of crystallisation in sodium carbonate crystals, $Na_2CO_3 \cdot xH_2O$

When sodium carbonate is crystallised from solution, it forms crystals of hydrated sodium carbonate, $Na_2CO_3 \cdot xH_2O$.

In this question, you will perform a titration. The data from the titration will be used to calculate:
- the concentration of Na_2CO_3 in **FA 5**
- the M_r of $Na_2CO_3 \cdot xH_2O$ and hence the value of x.

FA 5 contains 10.8 g dm^{-3} of $Na_2CO_3 \cdot xH_2O$ in deionised water.
FA 6 is 0.100 mol dm^{-3} hydrochloric acid, HC*l*.
Solution I is methyl orange indicator.

(a) (i) Titration of FA 5 against FA 6

1. Fill the burette with **FA 6**.
2. Use the pipette to transfer 25.0 cm^3 of **FA 5** into a 250 cm^3 conical flask.
3. Add a few drops of **Solution I** to the same conical flask.
4. Titrate the mixture in the conical flask until the end-point is reached.
5. Record your titration results, to an appropriate level of precision, in the space provided below.
6. Repeat points 2 to 5 until consistent results are obtained.

Titration results

Titration	1	2
Final burette reading / cm^3	19.20	38.50
Initial burette reading / cm^3	0.00	19.20
Volume of FA6 used / cm^3	19.20	19.30

[3]

(ii) From your titrations, obtain a suitable volume of **FA 6** to be used in your calculations. Show clearly how you obtained this volume.

Average vol. of FA6 used = $\frac{19.20 + 19.30}{2}$ = 19.25 cm^3

volume of **FA 6** = ... cm^3 [4]

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(b) (i) The equation for the reaction in the titration is shown.

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2 + H_2O$$

Calculate the concentration of Na_2CO_3 in **FA 5**.

Amount of HC*l* used = $\frac{19.25}{1000} \times 0.100 = 1.925 \times 10^{-3}$ mol

Amount of Na_2CO_3 in 25.0 cm^3 of FA5 = ½ × $1.925 \times 10^{-3} = 9.625 \times 10^{-4}$ mol

Concentration of Na_2CO_3 in FA5 = $\frac{9.625 \times 10^{-4}}{(25.0/1000)}$ = 0.0385 mol dm^{-3}

concentration of Na_2CO_3 = ... mol dm^{-3} [1]

(ii) Determine the M_r of $Na_2CO_3 \cdot xH_2O$ and hence the value of x.
You **must** show your working.
[A_r: H, 1.0 C, 12.0 O, 16.0 Na, 23.0]

M_r of $Na_2CO_3 \cdot xH_2O$ = $\frac{10.8}{0.0385}$ = 280.5 (1d.p.)

M_r of $Na_2CO_3 \cdot xH_2O$ = ...

M_r of $Na_2CO_3 \cdot xH_2O = 280.5$

$23.0 \times 2 + 12.0 + 16.0 \times 3 + (18.0x) = 280.5$

$18.0x = 174.5$

$x = 9.70 \approx 10$ (whole number)

x = ...
[2]

(c) (i) The reaction in the titration produces carbon dioxide. Some carbon dioxide dissolves in the solution. Dissolved carbon dioxide is acidic.

Methyl orange indicator changes colour over a pH range of 3.1 to 4.4.

Explain why the dissolved carbon dioxide does **not** decrease the end-point of the titration.

CO_2 dissolves in H_2O to give H_2CO_3, which is weakly acidic. It is likely that H_2CO_3 solution has a pH of above 4.4, and hence, would not cause the methyl orange indicator to change colour before the acid-base reaction is completed. The indicator only changes colour when one excess drop of strong acid HC*l* causes the pH to go below 4.4. [1]

(ii) A student follows the procedure described using sodium carbonate crystals with a different amount of water of crystallisation. The student calculates the value of x in these crystals to be 6.94. The formula of these crystals is $Na_2CO_3 \cdot 7H_2O$.

Calculate the difference between the actual value and the student's calculated value as a percentage. This is the percentage error.

Difference = 6.94 – 7 = −0.06

$$\% \text{ error} = \frac{-0.06}{7} \times 100 = -0.857\%$$

Percentage error = ...% [1]

(iii) The student calculates that the maximum total percentage error of the whole procedure is 1.08%.

Did the student perform the experiment well? Explain your answer.

Yes, since the student's percentage error of result is lower than the total percentage error of the whole procedure. [1]

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(d) (i) Planning

The amount of water of crystallisation in sodium carbonate crystals can also be determined gravimetrically (by weighing).

The water of crystallisation in sodium carbonate crystals can be removed by heating. Sodium carbonate crystals do not decompose at temperatures achievable by a Bunsen flame.

Plan an investigation to determine the value of x in sodium carbonate crystals, $Na_2CO_3 \cdot xH_2O$, gravimetrically.

You may assume that you are provided with:

- approximately 10 g of sodium carbonate crystals, $Na_2CO_3 \cdot xH_2O$
- 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 (you may find it useful to label measurements in your plan as M1, M2 etc.)
- how you would ensure that an **accurate** and **reliable** value of x is obtained.

Procedure:

1. Weigh a dry, empty boiling tube using an electronic balance and record its mass, M1

2. Weigh accurately about 4.0 g of the sodium carbonate crystals into the boiling tube.

Record the total mass of the boiling tube and its contents, M2.

3. Heat the boiling tube and its contents gently first, then strongly for about 5 minutes.

4. Tap the boiling tube intermittently to expose all solid particles to the heat.

5. Heat along the length of the boiling tube to prevent condensation of water vapour on the cooler surface of the boiling tube.

6. After heating, place the hot boiling tube on a heat proof mat to cool.

7. Once the boiling tube has cooled down, weigh the boiling tube containing the residue and record its mass, M3.

8. Repeat the steps 3 to 7 (heat-cool-weigh process) until mass of boiling tube and residue is consistent within ±0.010 g. Record all the mass recordings.

9. To ensure reliability, repeat the experiment to collect another set of data to calculate the value of x.

Mass of empty boiling tube / g	M1
Mass of boiling tube + solid / g	M2
Mass of boiling tube + solid after 1st heating / g	M3
Mass of boiling tube + solid after 2nd heating / g	M4 (±0.010 from M3)

[6]

(ii) Describe how you would use the labelled measurements in your answer to **(d)(i)** to calculate a value of x.
[A_r: H, 1.0 C, 12.0 O, 16.0 Na, 23.0]

$Na_2CO_3 \bullet xH_2O \longrightarrow Na_2CO_3 + xH_2O$

Mass of H_2O lost = M2 – M4

Mass of anhydrous Na_2CO_3 = M4 – M1

Amount of H_2O lost = $\frac{M2 - M4}{18.0}$

Amount of anhydrous Na_2CO_3 = $\frac{M4 - M1}{106.0}$

$x = \frac{\text{amount of } H_2O}{\text{amount of anhydrous } Na_2CO_3}$

[3]

[Total: 22]

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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

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(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 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

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