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

1 Determination of the M_r of a carbonate salt

FA 1 is a solid carbonate, **X**CO_3.

FA 2 is a solution containing 1.00 mol dm^{-3} of hydrochloric acid, HCl.

FA 3 is a solution of NaOH of concentration 0.100 mol dm^{-3}.

In this question, you will perform a titration. The data from this titration will be used to determine

- the amount of CO_3^{2-} ions in the mass of **FA 1** used,
- the M_r of **X**CO_3 and hence the identity of the metal **X**.

(a) Titration of FA 4 against FA 3

(i) You are to determine the amount of carbonate ions, CO_3^{2-}, in **FA 1,** by back titration after some **FA 2** has been added to it. Carry out the procedure as listed below.

1. Weigh accurately between 1.25 – 1.35 g of **FA 1** into a clean and dry 150 cm^3 beaker. If you have used the TARE facility on the balance, indicate clearly in your recording.
2. Record your weighing in an appropriate format in the space provided on the next page.
3. Measure out 50 cm^3 of **FA 2** into a measuring cylinder. Pour this into the beaker containing **FA 1.** Stir thoroughly to ensure all of **FA 1** has dissolved.
4. Quantitatively transfer the mixture into a 250 cm^3 volumetric flask. Make up to the mark with distilled water. Shake to obtain a homogeneous solution. Label this solution **FA 4.**
5. Pipette 25.0 cm^3 of **FA 4** into a conical flask and add 2 drops of methyl orange indicator.
6. Fill the burette with **FA 3.**
7. Titrate **FA 4** with **FA 3.**
8. Repeat the titration as many times as you consider necessary to obtain accurate results.
9. Record your titration results in the space provided on the next page. Make certain that your recorded results show the precision of your working.

Results:

*For weighing of **FA 1***

Titration

[6]

(ii) From your titrations, obtain a suitable volume of **FA 3** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 3** = ………………….. [1]

(b) **(i)** Calculate the amount of excess acid pipetted into the conical flask.

amount of excess acid in the conical flask = …………………… [1]

(ii) Calculate the total amount of excess acid in the volumetric flask.

amount of excess acid in the volumetric flask = …………………… [1]

(iii) Hence calculate the amount of acid that has reacted with the carbonate that you have weighed out.

amount of acid reacted = …………………….. [1]

(iv) Use your answer in **b(iii)** to calculate the M_r of **X**CO_3.

M_r of **X**CO_3 = ……………………..

Deduce the identity of metal **X**.
Show your working.

[A_r: C, 12.0; O, 16.0; H, 1.0; Be, 9.0; Mg, 24.3; Ca, 40.1; Sr, 87.6; Ba, 137.3]

X is ………………………………… [3]

(c) Planning

Sodium carbonate, Na_2CO_3, does **not** decompose on heating with a Bunsen burner. Sodium hydrogencarbonate, $NaHCO_3$, decomposes on heating.

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

You are to design an experiment in which the percentage by mass of $NaHCO_3$ in a mixture of $NaHCO_3$ and Na_2CO_3 can be determined by heating and weighing alone.

The only apparatus available consists of:

a boiling tube and holder
a chemical balance
a Bunsen burner
a heat proof mat

You are to show how you would use the results of this experiment to determine the percentage by mass of $NaHCO_3$ in the mixture.

(i) Outline, step by step, the practical sequence for the method you would use to:

- make appropriate weighings,
- decompose the sodium hydrogencarbonate in the mixture by heating,
- ensure that the decomposition was complete.

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.... [3]

(ii) By considering the products of the decomposition, suggest a reason why a crucible, without a lid, might be more appropriate than a boiling tube for this experiment.

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(iii) Prepare a table to show the masses you would measure and record during the experiment. Include in your table any other masses you would calculate from the experimental results to enable you to determine the percentage by mass of $NaHCO_3$ in the mixture.

Insert in your table the letters **A**, **B**, **C** etc. to represent each mass. Use these letters to show how your calculated masses are obtained e.g. **B** – **A**.

[2]

(iv) Use the letters you have entered in **(c)(iii)** to show how you would process the results to find:

- the mass of $NaHCO_3$ in the mixture,

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

- the percentage by mass of $NaHCO_3$ in the mixture.

[2]

[Total: 21]

2 Determination of the concentration of a mixture of acids and the enthalpy change of neutralization

FA 5 is an aqueous solution prepared by mixing equal volumes of y mol dm^{-3} hydrochloric acid, HCl, and y mol dm^{-3} sulfuric acid, H_2SO_4.

FA 6 is 1.60 mol dm^{-3} sodium hydroxide, $NaOH$.

Take care as aqueous solutions of sodium hydroxide are corrosive.

When an acid is run into an alkali, an exothermic reaction takes place and the temperature of the mixture increases until the end–point is reached. If acid is added beyond the end–point, the temperature will decrease as no further reaction takes place and the acid is at a lower temperature than the mixture.

You are to follow the neutralisation of the acids in **FA 5** by measuring the temperature as volumes of **FA 5** are added in regular portions from a burette to a fixed volume of **FA 6** placed in a Styrofoam cup.

The data obtained will enable you to determine the value of y and the enthalpy change of neutralization, ΔH_{neut}, of this acid–base reaction.

In an appropriate format in the space provided on the next page, prepare a table in which you may record the following after each addition of **FA 5**:

- Total volume of **FA 5** added from the burette up to the point in time
- Total volume of solution in the cup (V_{cup})
- Temperature measured (T)

You also need to calculate the corresponding values of:

- $\Delta T = T - T_0$, where T_0 is the initial temperature of **FA 6**
- ($V_{cup} \times \Delta T$) to 3 significant figures

1. Fill the burette to 0.00 cm^3 with **FA 5**.

2. Place the Styrofoam cup in a 250 cm^3 beaker to provide support for the cup. Use the measuring cylinder to place 40.0 cm^3 of **FA 6** into the cup and measure the steady temperature of the alkali, T_0.

3. Run 5.00 cm^3 of **FA 5** from the burette into the cup, stir the solution with the thermometer and record the maximum temperature, T.

4. **Immediately** run a further 5.00 cm^3 of **FA 5** from the burette into the cup, stir and record the maximum temperature as before.

5. Continue the addition of **FA 5** in 5.00 cm^3 portions and record the maximum or minimum temperature reached after each addition. Do this until a total of 45.00 cm^3 of solution have been run from the burette.

(a) (i) Experimental results

[3]

(ii) On the grid provided below, plot (V_{cup} × ΔT) against the total volume of **FA 5** added.

Draw a line of best fit for the points before the maximum value of (V_{cup} × ΔT).

Draw a second line of best fit for the points after the maximum value.

Extrapolate both lines until they meet.

[2]

(iii) Read from your graph the maximum value of (V_{cup} × ΔT) and the end–point volume of the titration.

maximum value of (V_{cup} × ΔT) = ……………………..

end–point volume = …………………….. [3]

(b) **(i)** Calculate the concentration of hydrogen ions in **FA 5**.

concentration of hydrogen ions = …………………….. [1]

(ii) Hence, determine the value of ***y***, which is the concentration of either HC*l* or H_2SO_4 used to prepare **FA 5**.

y = ……………………… [1]

(c) **(i)** Determine the enthalpy change of neutralization of this reaction, ΔH_{neut}.

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

ΔH_{neut} = …………………….. [3]

(ii) Student **A** carefully performed the same experiment and correctly processed the data using the same method. His calculated value of ΔH_{neut} is –65.0 kJ mol^{-1} while the published value for this enthalpy change is –57.7 kJ mol^{-1}.

The specific heat capacity of the Styrofoam cup has not been taken into consideration in calculating ΔH_{neut}. Explain whether this omission could have been the reason for the discrepancy between the value obtained by Student **A** and the one published.

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(iii) Student **B** performed the same experiment but chose to plot ΔT instead of ($V_{cup} \times \Delta T$) on the y–axis.

He also drew and extrapolated two straight lines through the plotted points to do the calculations.

Suggest why his method is likely to yield **less** accurate results.

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[Total: 16]

3 Inorganic Analysis

(a) You are provided with the solid **FA 7** which contains one cation and one anion from the ions listed in the **Qualitative Analysis Notes**, and the solution **FA 8**.

You are to perform the tests below to identify the ions present in **FA 7** and to suggest the nature of the compound in **FA 8**.

If the evolution of a gas is observed at any stage, the gas should be tested and identified. Details of the test carried out, the observations of the test and the identity of the gas should be given with the observations.

If it appears that no reaction has taken place this should be clearly recorded.

Carry out the following tests on **FA 7** and **FA 8** and complete the table to show your observations.

	Test	Observations
(i)	To a spatula–full of **FA 7** in a test–tube add 3 cm depth of dilute sulfuric acid. Warm, and when the reaction has stopped, filter the mixture. Retain the filtrate for tests **(iii)** and **(iv)**.	
(ii)	Place a spatula–full of **FA 7** in a test–tube and heat strongly. Leave the tube to cool. Retain the residue for test **(viii)**.	
(iii)	To a 1 cm depth of the filtrate from **(i)** in a test–tube, add aqueous sodium hydroxide drop by drop until 1 cm depth of the reagent has been added. Then add a further 1 cm depth of the aqueous sodium hydroxide.	
	Swirl the test-tube and leave to stand for 2–3 minutes.	
(iv)	To a 1 cm depth of the filtrate from **(i)** in a test–tube, add aqueous ammonia drop by drop until 1 cm depth of the reagent has been added. Then add a further 1 cm depth of the aqueous ammonia.	
	Then **cautiously** add 1 cm depth of **FA 8**.	

(v)	Place 2 cm depth of aqueous potassium iodide in a test–tube and add 2 cm depth of **FA 8**.	
	Then add 2 cm depth of dilute sulfuric acid.	
(vi)	To a 1 cm depth of **FA 8** in a test–tube, add 1 cm depth of dilute sulfuric acid and 2 cm depth of potassium manganate(VII). Observe for 2 minutes or until no further change occurs.	
(vii)	To a 1 cm depth of aqueous sodium sulfite in a test–tube, add a few drops of aqueous barium nitrate.	
	Then add 2 cm depth of dilute nitric acid.	
	Immediately add 1 cm depth of **FA 8**.	
(viii)	Transfer a portion of the residue from test **(ii)** into a test–tube and add 2 cm depth of **FA 8**.	

[9]

(b) **(i)** From your observations, identify the cation and anion present in **FA 7**. Quote evidence from the table to support your conclusions.

Cation ………………………………

Evidence ……………………………………………………………………..

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Anion ………………………………

Evidence ……………………………………………………………………..

…………………………………………………………………… [2]

(ii) In tests **(a)(v)** and **(vii)**, **FA 8** behaves as ……………………………………...

In test **(a)(vi)**, **FA 8** behaves as …………………………………………….. [1]

(c) **Planning**

One of the transition metals, vanadium, was named after the Scandinavian goddess of beauty and fertility, Vanadis (Freya) due to the wide range of colours found in vanadium compounds.

Starting with an aqueous solution of VO_2^+, you are to devise a plan which will enable you to determine the colour exhibited by two other ions containing vanadium, VO^{2+} and V^{3+}. Your plan should only involve simple test–tube reactions.

The standard electrode potentials of the following ions are as shown:

$$VO_2^+ + 2H^+ + e^- \rightleftharpoons H_2O + VO^{2+} \qquad E^\ominus = +1.00\ V$$
$$VO^{2+} + 2H^+ + e^- \rightleftharpoons H_2O + V^{3+} \qquad E^\ominus = +0.34\ V$$
$$V^{3+} + e^- \rightleftharpoons V^{2+} \qquad E^\ominus = -0.26\ V$$

There are four reducing agents available as shown:

$$Zn^{2+} + 2e^- \rightleftharpoons Zn \qquad E^\ominus = -0.76\ V$$
$$Sn^{2+} + 2e^- \rightleftharpoons Sn \qquad E^\ominus = -0.14\ V$$
$$O_2 + 2H^+ + 2e^- \rightleftharpoons H_2O_2 \qquad E^\ominus = +0.68\ V$$
$$Fe^{3+} + e^- \rightleftharpoons Fe^{2+} \qquad E^\ominus = +0.77\ V$$

(i) Explain which reducing agent(s) will **not** be suitable for determining the colours exhibited by VO^{2+} and V^{3+}.

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(ii) Complete the table by choosing a suitable reducing agent for determining the colour exhibited by VO^{2+} and V^{3+} respectively. Justify your choice by calculating relevant $E^{\ominus}_{cell}$ values.

	Reducing agent	Justification
$VO_2^+ \rightarrow VO^{2+}$		
$VO_2^+ \rightarrow V^{3+}$		

[4]

[Total: 18]