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CAMBRIDGE A LEVEL PROGRAMME

CHEMISTRY 9701

PRACTICAL MANUAL

(SEMESTER 2)

JULY 2012 INTAKE

BOOK 2

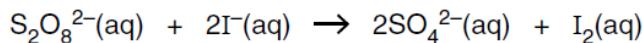
Name :

Class :

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Practical 1

- 1 You are required to investigate the effect of temperature on the rate of reaction of peroxydisulfate ions with iodide ions. Iodide ions are oxidised to iodine by peroxydisulfate ions.



FA 1 is aqueous potassium peroxydisulfate, $\text{K}_2\text{S}_2\text{O}_8$.

FA 2 is an aqueous solution containing a mixture of potassium iodide, KI, sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, and starch.

When **FA 1** and **FA 2** are mixed together the potassium peroxydisulfate reacts with the potassium iodide to make iodine. As soon as this iodine is formed, it reacts with the sodium thiosulfate and is turned back into iodide ions. Only when all the sodium thiosulfate has reacted does iodine remain in the solution. The solution then turns blue-black because of the presence of the starch indicator. The rate of reaction can be determined by the time it takes for a blue-black colour to first appear in the colourless mixture.

(a) Method

Read through the method and prepare a table on page 3 to record the initial and final temperatures and the reaction time for each experiment, before starting any practical work.

- Half-fill a 250cm^3 beaker with water to act as a water bath.
- Place it on a tripod and gauze and heat it with a Bunsen burner to about 65°C then remove the Bunsen. While your water is being heated continue with the following steps of the method.
- Fill the burette, labelled **FA 1**, with the aqueous potassium peroxydisulfate, **FA 1**.
- Fill the burette, labelled **FA 2**, with the mixture of solutions, **FA 2**.
- Measure 10.0cm^3 of **FA 1** into a boiling tube.
- Measure 10.0cm^3 of **FA 2** into a second boiling tube.
- Place both boiling tubes in the water bath.
- Clamp one of the tubes and place a thermometer in this tube.
- When the temperature of this solution has reached about 60°C , pour the contents of the second tube into the clamped tube. Start timing immediately, note the temperature and stir the mixture.
- Record this initial temperature.
- Stop timing as soon as the blue-black colour appears. Record this reaction time to the **nearest second** and record the final temperature.
- Repeat the experiment at decreasing temperatures as many times as necessary to generate data for plotting a graph. The experiment should not be performed at a temperature below about 30°C . The temperature of the water bath may be adjusted by adding cold water or by reheating. (Boiling tubes may be rinsed and reused.)

[5]

- (b) The rate of reaction for each experiment can be represented by the following.

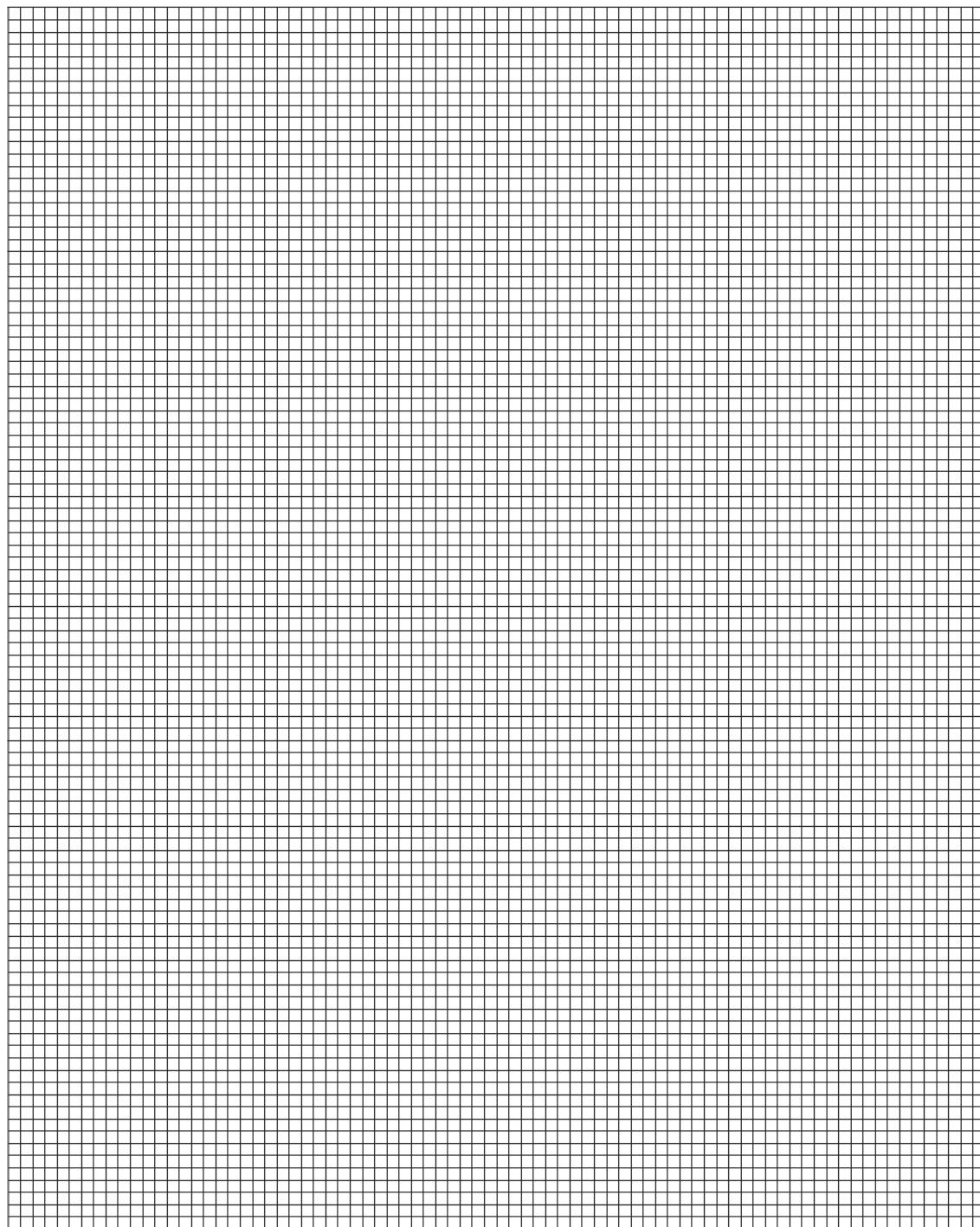
$$\text{‘rate’} = \frac{1000}{\text{reaction time in seconds}}$$

Complete the following table for each of your experiments. The mean temperature is the average of the initial and final temperature for the experiment.

mean temperature / °C	‘rate’

[6]

- (c) (i)** Using your values in **(b)**, plot a graph of 'rate' (y-axis) against mean temperature (x-axis). Choose suitable scales to allow you to extrapolate the graph to include the 'rate' at 20°C .



- (ii)** Use your graph to obtain a value for the 'rate' at 20°C .

'rate' at 20°C is [4]

- (d) It has been suggested that an increase in temperature of 10°C will double the rate of reaction. Use two pairs of temperatures from your graph to confirm or deny this statement.

.....
.....
.....

[2]

- (e) (i) Which of your experiments has the greatest percentage error in timing?

.....

- (ii) Calculate the percentage error in (i).

You may assume that the error in measuring the time for a reaction is ± 0.5 seconds.

error in (i) = % [2]

- (f) A student had difficulty in drawing a line of best fit. Identify a source of error in the experimental **procedure**. Do not include any errors involving the precision of apparatus.

.....
.....

[1]

- (g) Suggest a modification that could be used to reduce this error.

.....
.....

[1]

- (h) Using **FA 1**, **FA 2** and distilled water, describe how you could investigate the effect of **concentration** of potassium peroxydisulfate on the rate of reaction.

.....
.....
.....
.....

[3]

[Total: 24]

2 Qualitative analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.
Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test the full name or correct formula of the reagents must be given.

FA 3, FA 4 and FA 5 are aqueous solutions containing one cation and one anion. One of these solutions is a dilute acid and this is the only acid present.

- (a) (i) Select a single chemical reagent from those supplied which would allow you to identify the dilute acid. You may not use indicator paper.

reagent

- (ii) Use this reagent to test all three solutions and record your results in an appropriate form in the space below.

- (iii) From your observations in (ii), identify which solution is the dilute acid.

FA is the dilute acid.

[5]

- (b) The acid you have identified in (a)(iii) is dilute sulfuric acid.
Complete the following table.

<i>test</i>	<i>observations</i>
To 1 cm depth of FA 3 in a test-tube, add 1 cm depth of FA 4 , then add excess hydrochloric acid.	
To 1 cm depth of FA 4 in a test-tube, add 1 cm depth of FA 5 , then add excess hydrochloric acid.	
To 1 cm depth of FA 5 in a test-tube, add 1 cm depth of FA 3 , then add excess hydrochloric acid.	

[4]

- (c) For the two unidentified solutions, complete the following table.

<i>test</i>	<i>observations</i>	
	FA	FA
To 1 cm depth of unknown in a boiling tube, add NaOH(aq)		
warm the tube carefully		

[2]

- (d) From your observations in (a), (b) and (c), identify the ions present in the two solutions tested in (c), giving the relevant evidence for each. If you have not been able to identify one or more of the ions, explain why the evidence obtained was insufficient.

FA cation evidence

.....
.....

anion evidence

.....
.....

FA cation evidence

.....
.....

anion evidence

.....
.....

[4]

- (e) If one of the aqueous anions was a bromide, what would be the minimum evidence needed for its identification?

.....
.....
.....

[1]

[Total: 16]

Practical 2

1 Read through question 1 before starting any practical work.

You are provided with the following reagents.

- **FB 1**, hydrated copper(II) sulfate
- **FB 2**, aqueous copper(II) sulfate
- **FB 3**, aqueous sodium thiosulfate
- **FB 4**, aqueous potassium iodide
- **FB 5**, starch indicator solution

The formula of hydrated copper(II) sulfate is $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ where x shows the number of molecules of water of crystallisation present.

The value of x can be found by two different methods.

Method 1 involves heating to drive off water of crystallisation while **Method 2** uses a titration to determine the concentration of $\text{Cu}^{2+}(\text{aq})$.

(a) Method 1

- Weigh a crucible and record the mass.
- Add between 2.50 g and 2.70 g of **FB 1** and record the new mass.
- Place the crucible containing **FB 1** on a pipe clay triangle and heat gently for about four minutes with a Bunsen burner.
- Allow the crucible to cool. You should continue with **Method 2** while the crucible is cooling.
- Weigh the crucible and its contents.

Record all masses in the space below.

[3]

(b) Calculate the mass of water lost and the mass of copper(II) sulfate that remained after heating.

mass of water lost = g

mass of copper(II) sulfate remaining = g
[1]

- (c) Use your answer to (b) to calculate how many moles of water were lost and the moles of copper(II) sulfate, CuSO_4 , remaining after heating.
Show all of your working.
[A_f : Cu, 63.5; H, 1.0; O, 16.0; S, 32.1]

The hydrated copper(II) sulfate contained mol of water
and mol of CuSO_4 . [2]

- (d) Use your answer to (c) to determine the value of x in the formula of hydrated copper(II) sulfate, $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$.

$$x = \dots \quad [2]$$

(e) **Method 2**

- Fill the burette with **FB 3**, aqueous sodium thiosulfate.
- Pipette 25.0cm^3 of **FB 2** into a conical flask and use a measuring cylinder to add 10cm^3 of **FB 4**.
- Titrate this solution with **FB 3** from the burette until the mixture becomes yellow-brown. Do **not** add too much **FB 3** at this stage.
- An off-white precipitate is also present in the flask and this will mask the colour of the solution.
- Add approximately 1cm^3 of **FB 5**. The solution will become blue-black as a starch iodine complex is formed.
- Continue the titration until the blue-black colour of the complex just disappears leaving the off-white precipitate.
- Perform sufficient further titrations to obtain accurate results.
- Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

Summary

25.0cm^3 of **FB 2** reacted with cm^3 of **FB 3**.

Show which results you used to obtain the value of the volume of **FB 3** by placing a tick (\checkmark) under the readings used in your results. [11]

- (f) (i) In **Method 1** a student was advised to carry out all weighings using the same balance. What type of error might be introduced if more than one balance was used?

.....

..... [1]

- (ii) In **Method 2**, 10 cm³ of **FB 4** was added during the titration process. Assume that the measuring cylinder used could be read to ± 0.5 cm³. Calculate the percentage error in the measurement of this volume.

..... % error [1]

- (g) **Method 1** is usually less accurate than **Method 2** for finding the value of x in the formula of hydrated copper(II) sulfate, CuSO₄. x H₂O.

A group of students carried out **Method 1** correctly but calculated a value of 4 for x . The true value for x is 5.

Suggest an error in the practical procedure of the experiment that could account for this difference.

.....

..... [1]

- (h) Suggest a modification that could be made to the practical procedure in **Method 1** to reduce this error.

Explain why this modification should give an answer nearer to 5.

modification

.....

explanation

..... [2]

[Total: 24]

2 In this question you should use information from the Qualitative Analysis Notes on pages 9 and 10.

(a) Solutions **FB 6**, **FB 7** and **FB 8** are known to be either chlorides or sulfates of aluminium, magnesium or calcium. The addition of aqueous sodium hydroxide and aqueous ammonia can be used to give information about the cation present.

Add NaOH(aq) and NH₃(aq) separately to each of the solutions **FB 6**, **FB 7** and **FB 8**.

Rinse and reuse test-tubes where possible.

Record both the tests and your observations in an appropriate form in the space below.

From your observations identify the solutions containing aluminium, magnesium and calcium ions. In each case give evidence to support your answer.

Solution contains the aluminium ion.

supporting evidence

Solution contains the magnesium ion.

supporting evidence

Solution contains the calcium ion.

supporting evidence [7]

- (b)** Choose a pair of reagents that, used together, would identify which solution or solutions contain(s) the sulfate ion.

Carry out the tests and record, in the space below, the reagents used and the observations made.

The sulfate ion is present in [2]

[Total: 9]

- 3 (a) You are to carry out the tests given in the table below on solid **FB 9**.

Record details of any gases that are released in the reactions. These gases should be identified by a test, **described in the appropriate part of the table**.
No additional tests should be attempted.

	<i>test</i>	<i>observations</i>
(i)	Place a spatula measure of FB 9 in the small hard-glass test-tube labelled FB 9 and heat the solid strongly.	
(ii)	To 1 cm depth of aqueous sodium hydroxide in a boiling-tube, add 1 spatula measure of FB 9 , then gently heat the mixture, do not boil Care is needed when heating aqueous sodium hydroxide.	
(iii)	To 1 cm depth of aqueous sodium hydroxide in a boiling-tube, add 1 spatula measure of FB 9 and a piece of aluminium foil, then gently heat the mixture. Care is needed when heating aqueous sodium hydroxide.	[4]

- (b) What elements **must** be present in **FB 9** to give the results you have obtained in test (i) and test (iii)?

..... [1]

- (c) What is the function of the aluminium foil in test (iii)?

..... [1]

- (d) **Do not carry out this test**

What would you expect to see if 1 cm depth of dilute hydrochloric acid was added to a spatula measure of **FB 9**?

..... [1]

[Total: 7]

Practical 3

- 1 You are to determine the concentration of aqueous copper(II) sulfate by titration. The concentration of Cu^{2+} ions in a solution can be found by reaction with an excess of aqueous iodide ions to produce iodine. The amount of iodine formed can be found by titration with thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$.

FB 1 is aqueous copper(II) sulfate, CuSO_4 .

FB 2 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FB 3 is aqueous potassium iodide, KI.

starch indicator

Read through the instructions carefully before starting any practical work.

(a) Method

- Fill the burette with **FB 2**.
- Pipette 25.0 cm^3 of **FB 1** into a conical flask.
- Use a measuring cylinder to add 10 cm^3 of **FB 3** into the conical flask.
- Titrate this mixture with **FB 2** until the colour of the mixture changes from brown to yellow-brown. An off-white precipitate will also be present in the flask throughout the titration.
- Add approximately 1 cm^3 of starch indicator.
- Continue the titration until the blue-black colour of the starch-iodine complex just disappears leaving the off-white precipitate.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FB 2** added in each accurate titration.

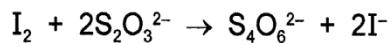
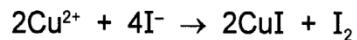
[6]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of FB 1 required cm³ of FB 2 [1]

(c) Calculations

The equations for the formation of iodine and its reaction with thiosulfate ions are given below.



Show your working and appropriate significant figures in the final answer to each step of your calculations.

- (i) Calculate the number of moles of thiosulfate ions, S₂O₃²⁻, present in the volume of FB 2 in (b).

$$\text{moles of S}_2\text{O}_3^{2-} = \dots \text{ mol}$$

- (ii) Using the equations above, deduce the number of moles of Cu²⁺ ions present in each 25.0 cm³.

$$\text{moles of Cu}^{2+} = \dots \text{ mol}$$

- (iii) Calculate the concentration, in mol dm⁻³, of copper(II) sulfate in FB 1.

$$\text{concentration of CuSO}_4 = \dots \text{ mol dm}^{-3}$$

[3]

- (d) Three students repeated the experiment but each obtained different values for the concentration of CuSO_4 .

The students each suggested possible improvements.

Student 1 suggested that a larger quantity of starch should be added.

Student 2 suggested that a larger volume of potassium iodide, **FB 3**, should be added.

Student 3 suggested that the contents of the conical flask should be filtered before titration.

Comment on the effectiveness of **each** of these possible improvements. Explain your answers.

Student 1

.....
.....

Student 2

.....
.....

Student 3

.....
.....

[2]

[Total: 12]

- 2** You are to determine the enthalpy change for the reaction between aqueous copper(II) sulfate and zinc. The enthalpy change of reaction can be found by measuring the temperature change when powdered zinc is added to aqueous copper(II) sulfate.

FB 4 is 1.10 mol dm⁻³ aqueous copper(II) sulfate, CuSO₄.
powdered zinc

(a) Method

- Weigh a 100 cm³ beaker.
- In the beaker weigh out between 2.1 g and 2.3 g of powdered zinc.
- Record the weighings and the mass of zinc in the space below.

mass of zinc used = g

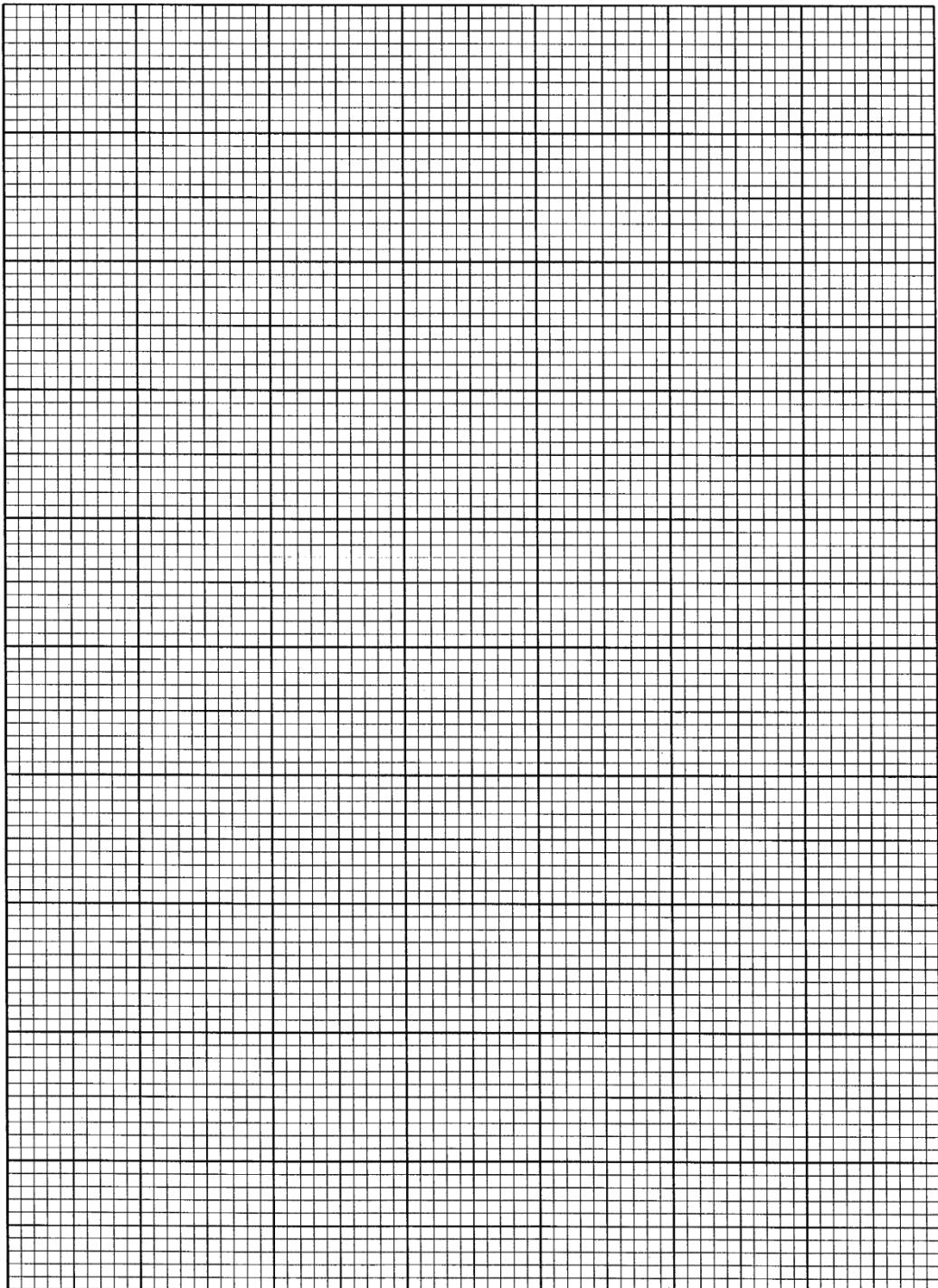
- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FB 4** into the plastic cup.
- Measure and record in the table below, the initial temperature of **FB 4** in the cup.
- Start the stop watch. Measure and record the temperature of **FB 4** in the cup after 1 minute, 2 minutes and 3 minutes.
- At time 3½ minutes, add the weighed zinc to **FB 4** in the cup and stir the mixture.
- From time 4 minutes, continue to stir the mixture and measure the temperature of the contents of the cup to complete the table.

Results

time/min	0	1	2	3	4	5	6	7	8	9	10	11	12
temperature/°C													

[2]

- (b) (i)** On the axes opposite, plot the temperature (y-axis) against time (x-axis). The temperature axis should allow you to include a point at least 5 °C greater than the maximum temperature recorded.



- (ii) Complete the graph to show how the temperature of the contents of the cup varies with time.
- Draw one straight line through the points between time 0 minutes and 3 minutes.
 - Draw one straight line through the points after the maximum was reached.
 - Extrapolate these two lines and draw a vertical line at time $3\frac{1}{2}$ minutes.

[4]

(c) Calculation

- (i) Use your graph to determine the change in temperature at 3½ minutes.

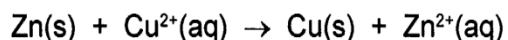
change in temperature = °C

- (ii) Calculate the heat energy produced in the reaction.

(You may assume that 4.3 J are required to raise the temperature of 1.0 cm³ of any solution by 1.0 °C.)

heat energy produced = J

- (iii) The reaction between zinc and aqueous copper(II) sulfate is a displacement reaction shown in the equation below.



From the mass of zinc added and the concentration of **FB 4**, show that the copper(II) sulfate was in excess in your reaction.

[A_r: Zn, 65.4]

- (iv) Assuming that the copper(II) sulfate was in excess, use your answer to (ii) to calculate the enthalpy change of the reaction between Zn(s) and Cu²⁺(aq).

Give you answer in kJ mol⁻¹ and include the relevant sign.

enthalpy change of reaction = kJ mol⁻¹
sign value

[6]

- (d) One source of error in this experiment is due to the accuracy to which the thermometer can be read.

What is the maximum error in a single temperature reading on a thermometer with graduations at 1 °C?

$$\text{maximum error} = \dots \text{°C}$$

Calculate the maximum percentage error when measuring a temperature **rise** of 12.0 °C.

$$\text{maximum percentage error} = \dots \% [2]$$

[Total: 14]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

You are provided with solutions **FB 5, FB 6, FB 7 and FB 8.**

FB 5 and FB 6 each contain a compound of a transition element.

Half fill a 250 cm³ beaker with water. Heat to approximately 80 °C, then stop heating and switch off the Bunsen burner. You will need this as a hot water bath on (b)(i). Continue work on (a) while the water heats.

(a) (i) Carry out the following tests on **FB 5**.

<i>test</i>	<i>observations</i>
To 1 cm depth of FB 5 in a test-tube, add aqueous ammonia.	
To 1 cm depth of FB 5 in a test-tube, add aqueous sodium hydroxide.	
To 1 cm depth of FB 5 in a test-tube, add aqueous barium chloride or aqueous barium nitrate then,	
add an excess of either hydrochloric acid or nitric acid.	

- (ii) From these tests, what conclusions, if any, can you reach about the identity of **FB 5**?

.....

[4]

- (b) (i) Carry out the following tests on **FB 6**.

<i>test</i>	<i>observations</i>
To 1 cm depth of FB 6 in a boiling tube, add 1 cm depth of FB 7 then,	
add 1 cm depth of ethanol. Place the boiling tube in the warm water bath and leave for a few minutes.	
To 1 cm depth of FB 6 in a test-tube, add 1 cm depth of FB 8 .	

- (ii) From these tests suggest identities for the following.

The anion in **FB 6** is

The cation in **FB 7** is

The cation in **FB 8** could be or

- (iii) Suggest a test to determine which of the two possible cations is present in **FB 8**.
Do not carry out this test.

.....

[7]

- (c) Using your conclusions about the possible identities of **FB 5** and **FB 8**, predict the result of mixing solutions of each.

Do not carry out this test.

Prediction

.....

[1]

- (d) Suggest what happened to the ethanol when it was warmed with the mixture of **FB 6** and **FB 7**.

..... [1]

- (e) You are to devise and carry out a test to confirm the identity of the cation in **FB 7**.

Record the test you use and the results of the test in the space below.

[1]

[Total: 14]

Practical 4

1 Read through question 1 before starting any practical work.

You are provided with the following reagents.

- weighing bottles/tubes labelled **FB 1**, **FB 2** and **FB 3**; each containing a different mass of sodium hydrogencarbonate, NaHCO_3
- additional solid sodium hydrogencarbonate (approximately 10 g)
- **FB 4**, 3.0 mol dm^{-3} hydrochloric acid, HCl

Use 2, 4, and 6 g respectively

The reaction of sodium hydrogencarbonate with hydrochloric acid is endothermic.

By measuring the maximum temperature decrease when the different masses of sodium hydrogencarbonate react with hydrochloric acid you are to determine the enthalpy change of neutralisation for 1 mol of NaHCO_3 with HCl .

(a) Method

- Weigh the bottle/tube containing the sodium hydrogencarbonate labelled **FB 1**.
- Support the plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 30 cm^3 of **FB 4** into the plastic cup.
- Place the thermometer in the acid in the plastic cup and record the steady temperature of the acid.
- Add the contents of the weighed tube, **FB 1**, to the acid in the plastic cup, a little at a time with constant stirring.
- You should add the solid as quickly as possible – taking care to minimise any acid spray from the plastic cup.
Avoid breathing any fumes from the experiment.
- Record the minimum temperature obtained in the reaction.
- Reweigh the emptied tube, **FB 1**, containing any remaining solid that was not tipped from the tube.
- Empty and rinse the plastic cup. Rinse the thermometer. Shake dry the plastic cup.
- Repeat the experiment using tubes labelled **FB 2** and **FB 3**. In each experiment use 30 cm^3 of **FB 4**.

Carry out **two further experiments**.

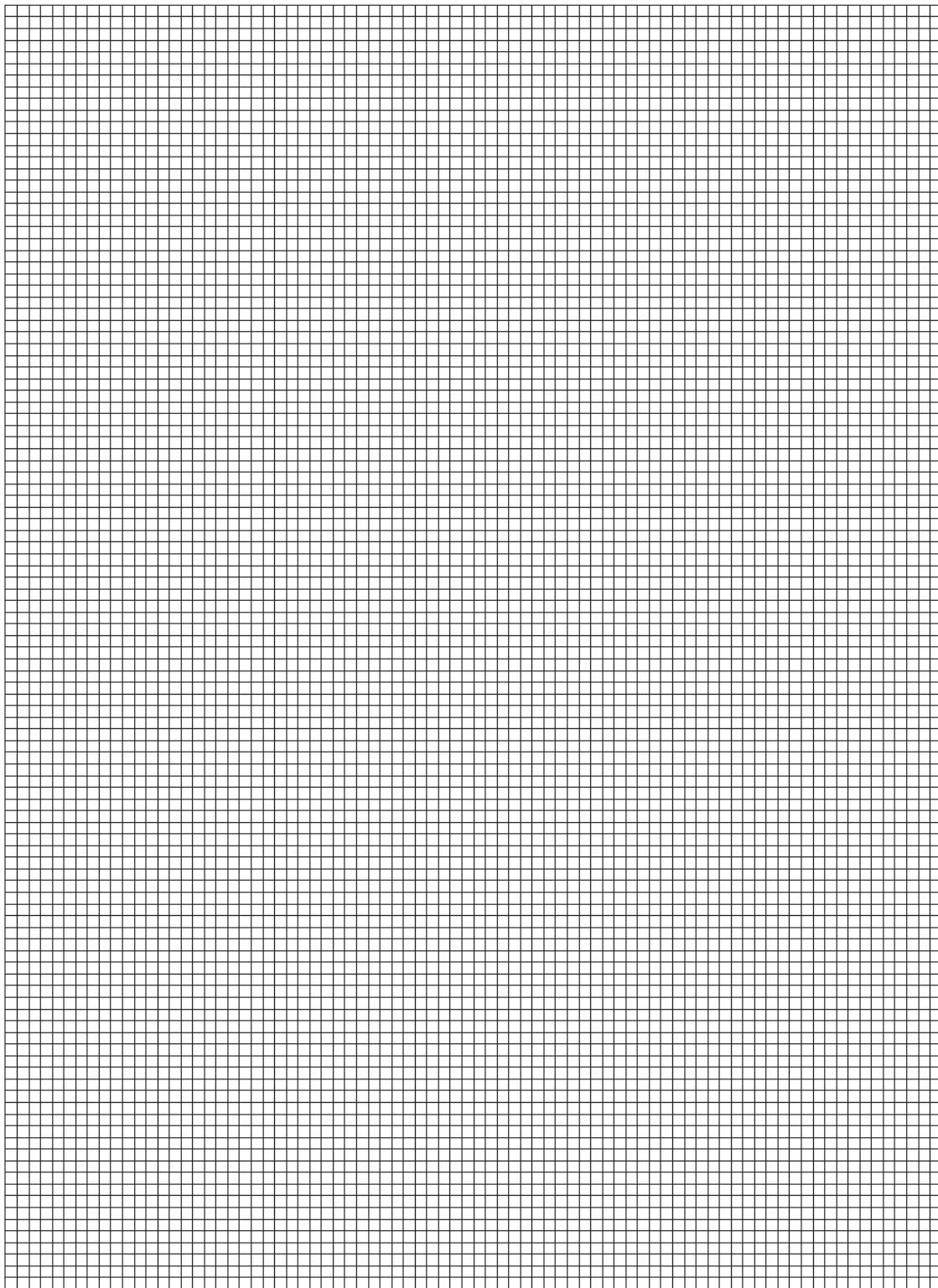
Using the empty weighing bottles/tubes, labelled **FB 5** and **FB 6**, weigh two further masses of sodium hydrogencarbonate. Choose masses to enable you to plot an appropriate graph of temperature change against mass of sodium hydrogencarbonate.

Results

Record your results in an appropriate form showing, for each experiment, the measurements of mass and temperature and the calculated temperature fall.

[9]

- (b) Use the grid below to plot a graph of decrease in temperature (*y*-axis) against the mass of sodium hydrogencarbonate added (*x*-axis).
Draw a line of best fit through the plotted points.
You should consider if the best-fit line passes through the origin (0,0) of the graph.



[4]

- (c) Explain why the mass of NaHCO_3 is plotted on the *x-axis* rather than on the *y-axis*.

.....
..... [1]

- (d) Construct the balanced equation for the reaction of NaHCO_3 with hydrochloric acid.

..... [1]

- (e) Calculate the gradient of your graph.

Show all of your working clearly, both construction lines on the graph and working in the calculation.

[3]

- (f) Although there is insufficient acid in 30cm^3 of **FB 4** to neutralise 1 mol of NaHCO_3 it is possible to calculate the theoretical fall in temperature for this reaction.

Use your answer from (e) to calculate this value.

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

The theoretical fall in temperature for 1 mol of NaHCO_3 = $^\circ\text{C}$ [1]

- (g) Calculate the theoretical enthalpy change for the neutralisation of 1 mol of NaHCO_3 by hydrochloric acid. Give your answer in kJmol^{-1} and include the correct sign for the reaction.

[4.3J are absorbed or released when the temperature of 1cm^3 of solution changes by $1\text{ }^\circ\text{C}$.]

ΔH = kJmol^{-1} [2]

- (h) Suggest **two** ways in which your apparatus could be modified to reduce transfer of heat from the surroundings to the solution in the apparatus.

modification 1

.....

modification 2

..... [1]

- (i) State and explain why the experiment would be more accurate if the volumes of **FB 4** had been measured using a burette instead of a measuring cylinder.

.....

..... [1]

- (j) The mass of NaHCO_3 used in a further experiment and its associated temperature change are shown in the tables below.

- The mass was obtained on a balance reading to 1 decimal place.
- The thermometer used was graduated at 1°C .

Complete the table to show the errors in these results.

mass of NaHCO_3	5.6 g
maximum error in a single balance reading	$\pm \dots \dots \dots$ g
% error in measured mass %

temperature change	-12.0°C
maximum error in a single thermometer reading	$\pm \dots \dots \dots ^\circ\text{C}$
% error in temperature change %

[2]

- (k) Two students add 6.0 g of sodium carbonate to 50.00 cm^3 of 2.0 mol dm^{-3} hydrochloric acid. Each student repeats the experiment a number of times.

The thermometer readings and temperature changes obtained consistently by each student are shown below.

	initial temperature / $^\circ\text{C}$	final temperature / $^\circ\text{C}$	temperature rise / $^\circ\text{C}$
student 1	20.0	28.0	8.0
student 2	19.0	27.0	8.0

Suggest the type of error shown by these results.

..... [1]

[Total: 26]

- 2 **FB 7** and **FB 8** are aqueous solutions of salts. One of these contains **two** cations and one anion.
The other contains one cation and one anion.
Both **FB 7** and **FB 8** have a common cation.

You will carry out tests to deduce the following.

- the cations present in each solution
- whether a sulfate ion is present in either solution

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate and the colour of the precipitate

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed directly with a Bunsen burner a boiling-tube MUST be used.

Rinse and reuse test-tubes where possible.

- (a) Use information from the Qualitative Analysis Notes on page 11 to select a pair of reagents that, **used together**, determine whether a sulfate ion is present in either solution.

The reagents are

followed by

[1]

- (b) Use your chosen reagents to carry out tests on **FB 7** and **FB 8**.
Record your results in an appropriate form in the space below.

[2]

- (c) From your observations in (b) show with a tick which of the following statements is true.

FB 7 contains the sulfate ion	
FB 8 contains the sulfate ion	
neither solution contains the sulfate ion	

Explain the evidence that supports your conclusion.

..... [1]

- (d) Carry out the following tests on the solutions **FB 7** and **FB 8**.
Record your observations below.

test	observations	
	FB 7	FB 8
To 1 cm depth of solution in a boiling-tube, add 2 cm depth of aqueous sodium hydroxide; then		
warm the solution gently.		
Care is needed when heating aqueous sodium hydroxide.		
To 1 cm depth of solution in a test-tube, add 2 cm depth of aqueous ammonia.		

[3]

- (e) To 1 cm depth of **FB 7** in a test-tube add 1 cm depth of sodium hydroxide.
Leave to stand for a few minutes.

observation

..... [1]

- (f) From your observations in (d) and (e) you should be able to identify the common cation in the solutions and the second cation in **one** of the solutions.

The common cation present in both solutions is

The second cation contained in one of the solutions is

Explain how your observations support your conclusions for

- (i) the common cation,

.....
.....

- (ii) the second cation.

.....
.....

[1]

Read through the remainder of question 2 before starting further practical work.

Heat a half-full 250 cm³ beaker of water for use as a hot water-bath.

- (g) **FB 9, FB 10, FB 11 and FB 12** are organic compounds. Each contains one of the following different functional groups.
- primary alcohol
 - secondary alcohol
 - aldehyde
 - ketone

You are to react each of these compounds with some of the following reagents.

- acidified aqueous potassium dichromate(VI)
- 2,4-dinitrophenylhydrazine (2,4-DNPH) reagent
- ammoniacal silver nitrate (Tollens' reagent)

You are provided with the first two reagents. You must prepare the last of these reagents, Tollens' reagent, immediately before use. Follow the instructions in the box below.

To 2 cm depth of aqueous silver nitrate in a boiling-tube add ½ cm depth of aqueous sodium hydroxide. This will produce a brown precipitate of silver(I) oxide.
Add aqueous ammonia a little at a time, with continuous shaking, until the brown precipitate **just** dissolves. **Do not add an excess of aqueous ammonia.**

In each of the following tests add a few drops of the reagent to 1 cm depth of **FB 9**, **FB 10**, **FB 11** and **FB 12** in separate test-tubes.

In the tests using acidified potassium dichromate(VI) and Tollens' reagent, if no initial reaction is seen, warm that tube and its contents in your hot water-bath. There is no need to heat any tube to which you have added 2,4-DNPH reagent.

Do **not** heat any tube with a naked flame.

Record your results in the table below.

Do **not** carry out tests for the shaded boxes.

reagent	observations			
	FB 9	FB 10	FB 11	FB 12
acidified potassium dichromate(VI)				
2,4-DNPH reagent				
Tollens' reagent				

[3]

(h) State which of the solutions contain alcohols. Explain the observations leading to your conclusion.

FB and **FB** contain alcohols.

explanation

.....

State which solution contains the ketone. Explain the observations leading to your conclusion.

FB contains the ketone.

explanation

.....

[2]

[Total: 14]

Practical 5

You must prepare **Flask A** and **Flask B** in Question 2 before starting Question 1. Shake each flask periodically during the time you spend on Question 1.

- 1 **FB 1** is $0.125 \text{ mol dm}^{-3}$ sulfuric acid, H_2SO_4 .
FB 2 is an aqueous solution of sodium hydroxide, NaOH .

You are to determine the concentration, in mol dm^{-3} , of the sodium hydroxide in **FB 2**.

(a) **Method**

- Fill a burette with **FB 1**.
- Run between 45.50 cm^3 and 46.50 cm^3 of **FB 1** from the burette into the 250 cm^3 graduated (volumetric) flask, labelled **FB 3**.
- Make up to the mark with distilled water.
- Shake the flask to mix the solution.

In the space below record your burette readings and the volume of **FB 1** added to the graduated flask.

You are reminded to shake **Flask A** and **Flask B** periodically.

Titration

- Fill a second burette with **FB 2**.
- Pipette 25.0 cm^3 of **FB 3**, the diluted acid, into a conical flask.
- Add to the flask a few drops of phenolphthalein indicator.
- Place the flask on a white tile.
- Titrate the acid in the flask with **FB 2**.
At the end-point a “permanent” pink colour will remain in the solution.
- **Note:** The “permanent” pink colour will fade over several minutes as carbon dioxide is absorbed from the atmosphere.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results
- Record in a suitable form on page 3 all of your burette readings and the volume of **FB 2** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

You will require the burette containing FB 2 for Question 2.

[7]

- (b) From your accurate titration results obtain a suitable value to be used in your calculation. Show clearly how you have obtained this value.

25.0 cm³ of **FB 3** required cm³ of **FB 2**.
[1]

Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

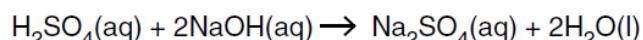
- (c) (i) Calculate how many moles of H₂SO₄ in **FB 1** were run from the burette into the 250 cm³ graduated, (volumetric) flask.

..... mol of H₂SO₄ were run from the burette into the graduated flask.

- (ii) Calculate how many moles of H₂SO₄ in **FB 3** were pipetted from the graduated flask into the conical flask in each titration.

..... mol of H₂SO₄ were pipetted into the conical flask.

- (iii) Calculate how many moles of NaOH reacted with the H₂SO₄ in (ii).



The H₂SO₄ in the titration flask reacted with mol of NaOH.

- (iv) Calculate the concentration, in mol dm^{-3} , of NaOH in **FB 2**.

The concentration of NaOH in **FB 2** is mol dm^{-3} .
[5]

- (d) The individual error in any burette reading is $\pm 0.05 \text{ cm}^3$.
Two students, A and B, record identical burette readings.

final burette reading	25.60 cm^3
initial burette reading	1.35 cm^3
volume added	24.25 cm^3

Explain the following.

- (i) The initial burette reading made by student A was 0.05 cm^3 greater than the true value but the volume added was exactly 24.25 cm^3 .

.....
.....

- (ii) The initial burette reading made by student B was 0.05 cm^3 less than the true value and the actual volume added was exactly 24.15 cm^3 .

.....
.....

[2]

- (e) In the instructions for the experiment you were told that the “permanent” pink colour at the end-point would fade over a few minutes as carbon dioxide is absorbed from the atmosphere.

- (i) Explain why absorption of carbon dioxide at the end-point would reverse the indicator colour change seen in the titration.

.....
.....

- (ii) Suggest a modification to the titration method, using the same indicator, that would overcome this problem.

.....
.....

[2]

[Total: 17]

- 2 **FB 4** is $0.050 \text{ mol dm}^{-3}$ sodium hydroxide solution.
FB 5 is $0.200 \text{ mol dm}^{-3}$ propanoic acid, $\text{C}_2\text{H}_5\text{CO}_2\text{H}$.
FB 6 is an organic liquid that does not mix with water.

Propanoic acid dissolves both in water and in the organic layer, **FB 6**. When an aqueous solution of the acid is shaken with **FB 6**, some of the acid transfers to the organic layer. The amount of acid remaining in the aqueous layer can be determined by titration with aqueous sodium hydroxide.

Preparation of the mixture in Flask A and in Flask B.

Flask A

- Use a measuring cylinder to place 50 cm^3 of **FB 5** into the stoppered flask labelled **Flask A**.
- Use a second measuring cylinder to add to the flask 40 cm^3 of **FB 6**, the organic liquid.
- Replace the stopper in the flask.

Flask B

- Use the first measuring cylinder to place 50 cm^3 of **FB 5** into the stoppered flask labelled **Flask B**.
- Use the second measuring cylinder to add to the flask 60 cm^3 of **FB 6**, the organic liquid.
- Replace the stopper in the flask.
- Shake both flasks vigorously for about 1 minute.
- **Leave the flasks on the workbench and start Question 1.**
- Shake the flasks for a further minute at intervals during the course of your work on another question.

(a) Titrations

For each flask follow the same procedure.

- Empty the burette containing **FB 2**.
- Rinse the burette thoroughly with **FB 4**.
- Fill the burette with **FB 4**.
- Ensure the two layers have separated – this should take no longer than 1 minute after shaking the flask.
- Pipette 10.0 cm^3 of the **lower** (aqueous) layer into a conical flask. Attach the pipette filler to the pipette before inserting it into the mixture, in order to close the top of the pipette to prevent any of the top (organic) layer from entering the pipette.
- Replace the stopper in the flask.
- Titrate the acid in the conical flask with **FB 4**, using phenolphthalein indicator, as in Question 1.
- **One titration will be sufficient for each experiment but take care to ensure that no errors are made during the procedure.**

Results

Record, in a single table below, the burette readings and volume of **FB 4** added, for each of **Flask A** and **Flask B**.

[4]

(b) Calculations

In these calculations make use of the following.

- The concentration of NaOH in **FB 4** is $0.050 \text{ mol dm}^{-3}$.
- 50 cm^3 of $0.200 \text{ mol dm}^{-3}$ propanoic acid, the volume of acid added to each flask, contains $0.010 \text{ mol } \text{C}_2\text{H}_5\text{CO}_2\text{H}$.
- $1 \text{ mol } \text{C}_2\text{H}_5\text{CO}_2\text{H}$ reacts with 1 mol NaOH .

- (i) Calculate the volume of **FB 4** that contains 0.010 mol NaOH .

This is the volume of **FB 4** that would have reacted with the propanoic acid in the 50 cm^3 of the aqueous layer, **before** shaking with the organic liquid.

Volume of **FB 4** = cm^3

- (ii) For each flask, use your titration result in (a) to calculate the volume of **FB 4** needed to react with the acid remaining in 50 cm^3 of the aqueous layer, **after** shaking with the organic liquid.

Flask A	Flask B
volume of FB 4 = cm^3	volume of FB 4 = cm^3

- (iii) The amount of propanoic acid transferred to the organic layer can be represented by the following.

(answer to (i) – answer to (ii))

For each flask evaluate this expression.

Flask A (answer to (i) – answer to (ii)) = cm^3

Flask B (answer to (i) – answer to (ii)) = cm^3

[2]

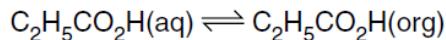
- (c) In which flask was most propanoic acid transferred to the organic layer?

Justify your answer.

.....
.....
.....

[1]

- (d) It is suggested that shaking the mixture leads to the following equilibrium being established.



Determine the equilibrium constant by evaluating the expressions in the following table.

- (i) Determine the equilibrium constant by evaluating the expressions in the following table. **Ignore units.**

Flask A	Flask B
$K_c = \frac{\text{answer (b)(iii)}}{\text{answer (b)(ii)}} \times 1.25$	$K_c = \frac{\text{answer (b)(iii)}}{\text{answer (b)(ii)}} \times 0.83$
$K_c = \dots$	$K_c = \dots$

- (ii) Explain whether or not your results support the idea that equilibrium has been established in each flask.

.....
.....
.....

[1]

[Total: 8]

- 3** **FB 7, FB 8 and FB 9** are aqueous solutions, each containing cations and anions from those listed on pages 11 and 12 in the Qualitative Analysis Notes.

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling-tube MUST be used.

Rinse and reuse test-tubes and boiling-tubes where possible.

- (a) (i) One or more of the solutions **FB 7, FB 8 and FB 9** are believed to contain the ammonium ion, NH_4^+ .

Suggest a reagent that would enable you to identify the presence of NH_4^+ and describe how you would use the reagent in an appropriate test.

reagent

test

.....
Use this reagent to test each of the solutions. Record your observations in the table below.

<i>solution</i>	<i>observation</i>
FB 7	
FB 8	
FB 9	

- (ii) One or more of the solutions contains the sulfate ion, SO_4^{2-} .
Select reagents that would enable you to identify the presence of SO_4^{2-} .
Show clearly, by describing how the reagents will be used, how you would distinguish SO_4^{2-} from the sulfite ion, SO_3^{2-} .

reagents

test

Use these reagents to test each of the solutions. Record your observations in the table below.

<i>solution</i>	<i>observation</i>
FB 7	
FB 8	
FB 9	

(iii) Conclusions

The ammonium ion, NH_4^+ , is present in

The sulfate ion, SO_4^{2-} , is present in

[5]

- (b)** Use aqueous sodium hydroxide and aqueous ammonia in separate tests to identify any cation (apart from NH_4^+) present in **FB 7**, **FB 8** and **FB 9**.

Present your results for each of the solutions in a suitable form below.

[4]

(c) Conclusion

Complete the following table.

Place a cross in any box where no cation has been identified.

<i>solution</i>	<i>cation</i>	<i>supporting evidence</i>
FB 7		
FB 8		
FB 9		

[1]

(d) Carry out the following tests on **FB 10**.

Observe carefully at each stage and record all of your observations in the table.

	<i>test</i>	<i>observations</i>
(i)	<p>Place 2 spatula measures of FB 10 in a dry, hard glass boiling-tube.</p> <p>Heat the solid gently at first, then strongly until no further change is seen.</p> <p>Retain the solid for use in (ii).</p>	
(ii)	<p>Tip the contents of the tube in (i) into a second boiling-tube.</p> <p>Add 2 cm depth of dilute hydrochloric acid a little at a time.</p> <p>Warm the tube and leave to stand.</p>	

[5]

[Total: 15]

Practical 6

- 1 You are to determine the percentage purity of a sample of calcium carbonate.

This experiment involves three steps.

In step one, you will react the impure calcium carbonate with an excess of acid.

In step two, you will carry out a titration to find the amount of acid you used in step one.

In step three, you will carry out a second titration to find how much (excess) acid remained following the reaction in step one.

Finally, you will use the values found in the three steps to calculate the percentage purity of the calcium carbonate.

Assume the impurity in the calcium carbonate will not react with acid or alkali.

FA 1 is $0.100 \text{ mol dm}^{-3}$ sodium hydroxide, NaOH .

FA 2 is approximately 1 mol dm^{-3} hydrochloric acid, HCl .

FA 3 is **FA 2** diluted by a factor of 10, approximately 0.1 mol dm^{-3} hydrochloric acid, HCl .

FA 4 is a sample of impure calcium carbonate, CaCO_3 .

methyl orange indicator

Read through the whole method before starting any practical work.

(a) Method

Step 1

- Fill the burette labelled **FA 2** with **FA 2**.
- Run 50.00 cm^3 of **FA 2** into a 250 cm^3 beaker.
- Weigh the tube containing the impure calcium carbonate, **FA 4**. **Use $\sim 2 \text{ g}$**
- Tip the contents of the tube, in small portions, into the acid to avoid acid spray. Stir the mixture and leave the stirring rod in the beaker.
- Reweigh the tube containing any residue.
- Record the weighings and the mass of **FA 4** added in a suitable form below.

Step 2

- Fill the burette labelled **FA 1** with **FA 1**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add methyl orange indicator.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 1** added in each accurate titration.

Step 3

- Stir the mixture from **Step 1** again to ensure that all the solid has dissolved.
- Transfer the solution to the 250 cm^3 graduated (volumetric) flask labelled **FA 5**.
- Rinse the beaker twice with a little distilled water and add the washings to the graduated flask.
- Make the solution up to 250 cm^3 with distilled water. Ensure that the contents of the flask are thoroughly mixed.
- Transfer 25.0 cm^3 of this solution, **FA 5**, into a second conical flask using a second pipette.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm^3 .

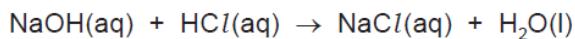
- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 1** added in each accurate titration.

[12]

Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

The equation for the reaction between sodium hydroxide and hydrochloric acid is shown below.



- (b) (i) From your accurate titration results in **Step 2**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FA 3** required cm³ of **FA 1**.

- (ii) Use your answer from (i) to calculate the number of moles of sodium hydroxide, **FA 1**, required to react with 25.0 cm³ of **FA 3** in **Step 2**.

moles of NaOH = mol

- (iii) Use your answer from (ii) to determine the number of moles of hydrochloric acid in 25.0 cm³ of **FA 3**.

moles of HCl in 25.0 cm³ of **FA 3** = mol

- (iv) **FA 3** was produced by diluting **FA 2**. Calculate the number of moles of hydrochloric acid in 50.00 cm³ of **FA 2**.

moles of HCl in 50.00 cm³ of **FA 2** = mol
[2]

- (c) (i) From your accurate titration results in **Step 3**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FA 5** required cm³ of **FA 1**.

- (ii) Use your answer from (i) to calculate the number of moles of sodium hydroxide, **FA 1**, required to react with 25.0 cm³ of **FA 5** in **Step 3**.

moles of NaOH = mol

- (iii) Use your answer from (ii) to determine the number of moles of hydrochloric acid in 25.0 cm³ of FA 5.

moles of HCl in 25.0 cm³ of FA 5 = mol

- (iv) Use your answer from (iii) to determine the number of moles of hydrochloric acid in 250 cm³ of FA 5.

moles of HCl in 250 cm³ of FA 5 = mol
[4]

- (d) (i) Write an equation for the reaction between calcium carbonate and hydrochloric acid.

.....

- (ii) Calculate the number of moles of hydrochloric acid that reacted with calcium carbonate in FA 4 using the following expression.

$$\text{moles of HCl} = (\text{b})(\text{iv}) - (\text{c})(\text{iv})$$

$$= \text{mol}$$

- (iii) Use your answers from (i) and (ii) to calculate the mass of CaCO₃ in FA 4.

[A_r; C, 12.0; O, 16.0; Ca, 40.1]

(If you were unable to answer (d)(ii), you may assume that the number of moles of hydrochloric acid that reacted with calcium carbonate was 0.0351 mol.)

mass of CaCO₃ in FA 4 = g

- (iv) Calculate the percentage purity by mass of the calcium carbonate in FA 4.

The percentage purity by mass of calcium carbonate is %
[4]

- (e) (i) What is the maximum error in a single burette reading?

maximum error in a burette reading = cm³

- (ii) Calculate the maximum percentage error for one of your accurate titres in **Step 3**. Show your working.

maximum percentage error = %
[2]

- (f) A student decided to use a larger mass of **FA 4**. State and explain whether this alteration will improve the accuracy of the percentage purity obtained.

.....
..... [1]

[Total: 25]

2 Qualitative analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, described in the appropriate place in your observations.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube **MUST** be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test the full name or correct formula of the reagent must be given.

Half fill a 250 cm³ beaker with water. Heat to approximately 80 °C, then stop heating. You will need this as a hot water bath in (a)(viii).

- (a) Compounds **FA 6**, **FA 7** and **FA 8** are salts containing the same transition metal but in three different oxidation states. You are provided with solutions of **FA 6** and **FA 7** and a solid sample of **FA 8**. Carry out the experiments described below and record your observations in the table.

<i>test</i>	<i>observations</i>
(i) To 1 cm depth of FA 6 in a test-tube add about 5 cm depth of dilute sulfuric acid and mix the two solutions. Use this mixture for tests (ii) and (iii).	no observation required
(ii) To 1 cm depth of hydrogen peroxide in a test-tube add 10 drops of acidified FA 6 from (i).	
(iii) To 1 cm depth of potassium iodide in a test-tube add 1 cm depth of acidified FA 6 from (i).	

<i>test</i>	<i>observations</i>
(iv) To 1 cm depth of FA 7 in a test-tube add 1 cm depth of aqueous sodium hydroxide and allow to stand for a few minutes.	
(v) To 1 cm depth of FA 7 in a test-tube add aqueous ammonia until in excess.	
(vi) To 1 cm depth of FA 7 in a test-tube add 1 cm depth of FA 6 and allow to stand for a few minutes.	
(vii) To 1 cm depth of hydrogen peroxide in a test-tube add a small spatula measure of FA 8.	
(viii) Place a small spatula measure of FA 8 in a test-tube and add about 10 drops of concentrated hydrochloric acid with care . Place the tube in the hot water bath. Test any gas produced with damp litmus paper. When you have made your observations, fill the test-tube with cold water to stop any further reaction.	

[6]

- (b) (i) From your observations in (a) suggest the identity of the transition metal contained in FA 6, FA 7 and FA 8.

The transition metal present in the three compounds is

Explain how your observations support your conclusion.

.....

.....

.....

- (ii) Suggest the type of reaction occurring in (a)(iii).

.....

- (iii) Give the oxidation state of the transition metal in FA 7.

.....

The oxidation state of the transition metal in FA 6 is +7.

Suggest an oxidation state of the transition metal in the product formed in (a)(vi).

.....

[3]

- (c) Aqueous solutions FA 9 and FA 10 both contain halide ions.

- (i) Use the Qualitative Analysis Notes on page 12 to select two reagents which, **used together**, identify the halide ions in FA 9 and FA 10.

The first reagent is

and this is followed by

- (ii) Use your chosen reagents to carry out tests on FA 9 and FA 10.
Record your results in an appropriate form in the space below.

- (iii) From the results of the tests in (ii), state which halide each solution contains.

FA 9 contains

FA 10 contains

- (iv) Halides can also be identified by reaction with concentrated sulfuric acid. The acid can act as an oxidising agent.

State what you would expect to see if concentrated sulfuric acid was added to a solid sample of **FA 9** and **FA 10**.

Do not carry out these experiments.

expected observation with **FA 9**

.....

expected observation with **FA 10**

.....

- (v) Solutions containing the copper(II) ion react with concentrated hydrochloric acid.

To a 1 cm depth of aqueous copper(II) sulfate in a test-tube, add an equal volume of concentrated hydrochloric acid **with care**.

observation

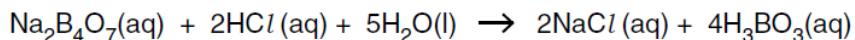
[6]

[Total: 15]

Practical 7

You are to determine the percentage by mass of water in the borax crystals.

Borax reacts with hydrochloric acid according to the equation.



- 1 **FA 1** is an aqueous solution containing 38.10 g dm^{-3} of **borax** crystals. **Borax** has the formula, $\text{Na}_2\text{B}_4\text{O}_7 \cdot x\text{H}_2\text{O}$.
FA 2 is 1.00 mol dm^{-3} hydrochloric acid, **HCl**.

You are also provided with an indicator suitable for the titration of a strong acid and a weak base.

The indicator provided is

(a) Method

Dilution

- Fill the burette with **FA 2**.
- Run between 44.50 cm^3 and 45.50 cm^3 of **FA 2** from the burette into the 250 cm^3 graduated (volumetric) flask, labelled **FA 3**.
- Make the solution up to the mark with distilled water.
- Shake the flask to mix the solution of **FA 3**.

In the space below record your burette readings and the volume of **FA 2** added to the graduated flask.

Titration

- Fill a second burette with **FA 3**, the diluted hydrochloric acid.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Add to the flask a few drops of the indicator provided.
- Titrate the borax in the flask with **FA 3** until the appropriate colour change is observed for the end-point.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 3** added in each accurate titration.

[7]

- (b) From your accurate titration results obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FA 1** required cm³ of **FA 3**.
[1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

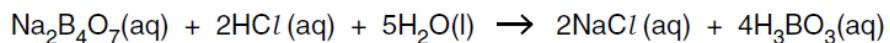
- (i) Calculate the concentration of hydrochloric acid, in mol dm⁻³, in the diluted solution, **FA 3**.

The concentration of HCl in **FA 3** was mol dm⁻³.

- (ii) Calculate how many moles of HCl were present in the volume of **FA 3** calculated in (b).

..... mol of HCl

- (iii) Calculate how many moles of $\text{Na}_2\text{B}_4\text{O}_7$ reacted with the HCl in (ii).



The HCl run from the burette reacted with mol of $\text{Na}_2\text{B}_4\text{O}_7$.

- (iv) Calculate the concentration, in g dm^{-3} , of $\text{Na}_2\text{B}_4\text{O}_7$ in FA 1.
[A_r : B, 10.8; O, 16.0; Na, 23.0]

The concentration of $\text{Na}_2\text{B}_4\text{O}_7$ in FA 1 is g dm^{-3} .

- (v) Use your answer to (iv) and the information at the start of question 1 to calculate the percentage by mass of water in the borax crystals, $\text{Na}_2\text{B}_4\text{O}_7 \cdot x\text{H}_2\text{O}$.
[A_r : H, 1.0; B, 10.8; O, 16.0; Na, 23.0]

Borax crystals contain % water.
[6]

- (d) The maximum error for a 25 cm^3 pipette commonly used in schools is $\pm 0.06 \text{ cm}^3$.
The maximum error in any single burette reading is $\pm 0.05 \text{ cm}^3$.

Calculate the maximum percentage error in each of the following.

- (i) The volume of FA 1 pipetted into the conical flask.

maximum percentage error in the pipetted volume = %.

- (ii) The titre volume calculated in (b).

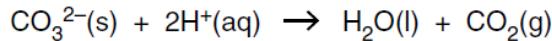
maximum percentage error in titre volume = %.
[1]

[Total: 15]

2 Read through the question carefully before starting any practical work.

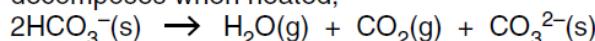
Sodium carbonate, Na_2CO_3 ,

- reacts exothermically with hydrochloric acid,
- does not decompose when heated,
- reacts with acids.

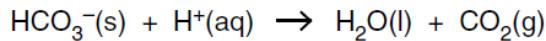


Sodium hydrogencarbonate, NaHCO_3 ,

- reacts endothermically with hydrochloric acid,
- decomposes when heated,



- reacts with acids.



You are to measure the temperature changes when samples of

- (i) sodium carbonate,
 - (ii) sodium hydrogencarbonate,
 - (iii) a mixture of sodium carbonate and sodium hydrogencarbonate,
- react with an excess of hydrochloric acid.

FA 4 is sodium carbonate, Na_2CO_3 .

FA 5 is sodium hydrogencarbonate, NaHCO_3 .

FA 6 is a mixture of sodium carbonate and sodium hydrogencarbonate.

FA 7 is 3.0 mol dm^{-3} hydrochloric acid, HCl .

Method

(a) sodium carbonate

- Support the plastic cup in a 250 cm^3 beaker.
- Use a measuring cylinder to transfer 50 cm^3 of **FA 7** into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled **FA 4**, containing Na_2CO_3 .
- **Carefully** tip the sodium carbonate from the weighed container **FA 4** into the hydrochloric acid in the plastic cup.

Note: There will be vigorous effervescence. Do not breathe the vapour. Add the solid in small portions with constant stirring using the thermometer.

- Record the highest temperature obtained.
- Reweigh the container **FA 4** with any residual sodium carbonate. Record the mass.
- Empty and rinse the plastic cup and dry it using a paper towel.

In the space at the top of the next page, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium carbonate, \mathbf{m}_1 , used in the experiment,
- the temperature rise, ΔT_1 .

[4]

Calculate the rise in temperature for each gram of sodium carbonate used in the experiment.

$$\frac{\Delta T_1}{m_1} = \boxed{+} \dots \text{sign} \quad \dots \text{value} \quad ^\circ\text{C g}^{-1}$$

(b) sodium hydrogencarbonate

- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of FA 7 into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of the container labelled FA 5, containing NaHCO₃.
- Carefully tip the sodium hydrogencarbonate from the weighed container FA 5 into the hydrochloric acid in the plastic cup.

Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.

- Record the lowest temperature obtained.
- Reweigh the container FA 5 with any residual sodium hydrogencarbonate. Record the mass.
- Empty and rinse the plastic cup and dry it using a paper towel.

In the space below, record, in an appropriate form,

- both balance readings and both temperature measurements,
- the mass of sodium hydrogencarbonate, m₂, used in the experiment,
- the temperature fall, ΔT₂.

[2]

Calculate the fall in temperature for each gram of sodium hydrogencarbonate used in the experiment.

$$\frac{\Delta T_2}{m_2} = \boxed{-} \dots \text{sign} \quad \dots \text{value} \quad ^\circ\text{C g}^{-1}$$

(c) mixture of sodium carbonate and sodium hydrogencarbonate

- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FA 7** into the plastic cup.
- Measure and record the temperature of the acid in the cup.
- Measure and record the mass of a clean, dry, weighing-bottle or tube.
- Add to the tube between **8.5 g and 9.5 g** of the mixture **FA 6**.
- Record the mass of the weighing-bottle or tube + **FA 6**.
- **Carefully** tip the weighed mixture into the hydrochloric acid in the plastic cup.
- **Note: There will be vigorous effervescence. Add the solid in small portions with constant stirring using the thermometer.**
- Record the highest or lowest temperature obtained.
- Reweigh the weighing-bottle or tube with any residual mixture. Record the mass.

In the space below, record, in an appropriate form,

- all balance readings and temperature measurements,
- the mass of the mixture, m_3 , used in the experiment,
- the temperature change, ΔT_3 .

Make certain that your recorded temperature change carries an appropriate sign.

[2]

(d) Transfer the following data from parts (a), (b) and (c).

$$(a) \frac{\Delta T_1}{m_1} = + \dots \text{ } ^\circ\text{C g}^{-1}$$

$$(b) \frac{\Delta T_2}{m_2} = - \dots \text{ } ^\circ\text{C g}^{-1}$$

$$(c) m_3 = \dots \text{ } \text{g} \quad \Delta T_3 = \boxed{} \dots \text{ } ^\circ\text{C}$$

sign

The masses of sodium carbonate and sodium hydrogencarbonate in the weighed sample of the mixture used in experiment (c) can be represented as follows.

$$\text{mass of sodium carbonate / g} = W$$

$$\text{mass of sodium hydrogencarbonate / g} = (m_3 - W)$$

Evaluate the following equation to determine a value for W .

$$[W \times \frac{\Delta T_1}{m_1}] + [(m_3 - W) \times \frac{\Delta T_2}{m_2}] = \Delta T_3$$

The mass of sodium carbonate was g.
[1]

- (e) Use the information at the beginning of question 2 to outline an alternative method that could be used in a school laboratory to find the mass of sodium carbonate and the mass of sodium hydrogencarbonate in the mixture FA 6.

.....
.....
.....
.....
.....

[1]

[Total: 10]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations**.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the full name or correct formula of the reagents must be given.

FA 8, FA 9 and FA 10 are aqueous solutions each containing a sodium cation and a single anion which could be a nitrite, a nitrate or a halide.

- (a) By reference to the Qualitative Analysis Notes on page 15, select a single reagent that would enable you to identify any solution containing the nitrite ion, NO_2^- .

reagent

Use **this reagent** to test each of the solutions. Record your observations in the table below. State clearly where no reaction has been observed.

<i>solution</i>	<i>observation</i>
FA 8	
FA 9	
FA 10	

[2]

- (b) By reference to the Qualitative Analysis Notes on page 15, select **one** reagent that would show that a halide ion is present.

reagent

Use **this reagent** to test each of the solutions.
Record your observations in an appropriate form in the space below.

Select **another** reagent to **identify or confirm** which halide ions are present in the solutions.

reagent

Tick the appropriate statement about the use of this reagent.

It is added to the tube already containing the first reagent.	
It is added to a fresh sample of solution.	

Use **this reagent** to identify or confirm which halide ions are present in the solutions and record your observations in an appropriate form in the space below.

[3]

- (c) From the results in (a) and (b) state which anions have been identified in the solutions.

Complete the following table.

Place a cross in any box if no anion has been identified.

<i>solution</i>	FA 8	FA 9	FA 10
<i>anion present</i>			

[1]

FA 11 and **FA 12** are aqueous solutions each containing one cation from those listed in the Qualitative Analysis Notes printed on page 14.

- (d) Use aqueous sodium hydroxide and aqueous ammonia in separate tests to identify the cation present in each of the solutions.
You will also require some of the solution, **FA 11**, for tests in (f).

Record the results of your experiments with sodium hydroxide and ammonia in an appropriate form in the space below.

[3]

(e) **Identification of the cations in FA 11 and FA 12**

Complete the table below.

<i>solution</i>	FA 11	FA 12
<i>cation present</i>		

What is the evidence **from your observations in (d)** that enables you to identify the cation present in each of the solutions?

The evidence supporting the conclusion for the cation in **FA 11** is

.....
.....

The evidence supporting the conclusion for the cation in **FA 12** is

.....
.....

[2]

(f) Complete the following table.

<i>test</i>	<i>observations</i>
(i) Pour 1 cm depth of FA 11 into a test-tube. Add 1 cm depth of aqueous potassium iodide. Divide this mixture into two parts for use in (ii) and (iii)	
(ii) To the first part of the mixture from (i) add a few drops of starch solution.	
(iii) To the second part of the mixture from (i) add aqueous sodium thiosulfate, a drop at a time, until no further change is observed.	

In part (i) and in part (iii) redox reactions have taken place.

Complete the table below to show the ion or molecule which has been oxidised and the ion or molecule which has been reduced in each of these reactions.

<i>reaction</i>	<i>the ion or molecule which has been</i>	
	<i>oxidised</i>	<i>reduced</i>
(i)		
(iii)		

[4]

[Total: 15]

Practical 8

- 1 **FA 1** is an aqueous solution of hydrochloric acid, HCl.
FA 2 is aqueous sodium hydroxide containing 10.00 g dm^{-3} NaOH.

You are to determine the concentration, in mol dm^{-3} , of the hydrochloric acid in **FA 1**.

(a) Method

- Fill a burette with **FA 2**.
- Pipette 10.0 cm^3 of **FA 1** into a conical flask.
- Add to the flask a few drops of the acid-base indicator provided.
- Place the flask on a white tile.
- Titrate the acid in the flask with **FA 2**.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record in a suitable form below all of your burette readings and the volume of **FA 2** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

[7]

- (b)** From your titration results obtain a suitable value to be used in your calculation.
Show clearly how you have obtained this value.

10.0 cm^3 of **FA 1** required cm^3 of **FA 2**.
[1]

Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

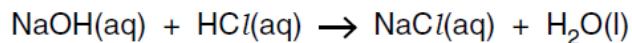
- (c) (i) Calculate the concentration, in mol dm^{-3} , of the sodium hydroxide in **FA 2**.
FA 2 contains 10.00 g dm^{-3} NaOH.
[A_r : H, 1.0; O, 16.0; Na, 23.0]

The concentration of sodium hydroxide in **FA 2** is mol dm^{-3} .

- (ii) Calculate how many moles of sodium hydroxide are contained in the volume recorded in (b).

..... mol of NaOH.

- (iii) Deduce how many moles of hydrochloric acid were pipetted into the conical flask and calculate the concentration, in mol dm^{-3} , of the hydrochloric acid in **FA 1**.



The concentration of the hydrochloric acid in **FA 1** is mol dm^{-3} .
[2]

[Total: 10]

2 FA 3 is crushed impure calcium carbonate, CaCO₃.

FA 4 is 0.500 mol dm⁻³ hydrochloric acid

FA 5 is 0.280 mol dm⁻³ sodium hydroxide.

You are to determine the percentage purity of calcium carbonate by dissolving a measured mass of FA 3 in a known volume of hydrochloric acid, which is in excess.

The hydrochloric acid remaining after all the calcium carbonate has dissolved can be determined by titration with aqueous sodium hydroxide, FA 4.

You may assume that any impurity present in the calcium carbonate does **not** react with hydrochloric acid.

(a) Method – Read through the instructions before starting any practical work.

- Weigh and record the mass of an empty boiling-tube.
- Add to the boiling-tube between 2.60 g and 2.80 g of FA 3.
- Reweigh the tube and its contents.
- In part (b) of the method you will tip the FA 3 into hydrochloric acid, then re-weigh the tube and any residual FA 3.

In the space below record, in an appropriate form, all of the balance readings and the mass of FA 3 used in the experiment.

[2]

(b) Method – Read through the instructions before starting any practical work.

- Pour approximately 150 cm³ of FA 4 into a 250 cm³ beaker.
- Add, a little at a time with constant stirring, the weighed FA 3 to the acid in the beaker.
- After each small addition stir until the effervescence has ceased and all the solid has dissolved.
- Reweigh the tube and any residual FA 3. Record the mass in (a).
- Transfer the solution in the beaker to the 250 cm³ graduated (volumetric) flask labelled FA 6.
- Rinse the beaker several times with a small amount of FA 4 and add the rinsings to the graduated flask.
- Make up the solution to the 250 cm³ mark by **adding FA 4, not water**.
- Shake the flask to obtain a uniform solution.

Titration

- Fill a burette with **FA 5**.
- Pipette 25.0 cm³ of **FA 6** from the graduated flask into a conical flask.
- Add to the flask a few drops of the acid-base indicator provided.
- Place the flask on a white tile.
- Titrate the acid in the flask with **FA 5**.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

The rough titre is cm³.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record in a suitable form below all of your burette readings and the volume of **FA 5** added in each titration.
- Make certain any recorded results show the precision of your practical work.

[2]

From your titration results obtain a suitable value to be used in your calculation.
Show clearly how you have obtained this value.

25.0 cm³ of **FA 6** required cm³ of **FA 5**.

(d) Calculations

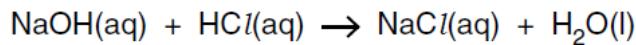
Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

Remember – FA 4 is $0.500 \text{ mol dm}^{-3}$ hydrochloric acid
FA 5 is $0.280 \text{ mol dm}^{-3}$ sodium hydroxide.

- (i) Calculate how many moles of sodium hydroxide are contained in the volume recorded in (c).

..... mol of NaOH

- (ii) Deduce how many moles of hydrochloric acid reacted with the sodium hydroxide in (i) and calculate how many moles of hydrochloric acid were present in the 250 cm^3 graduated flask labelled **FA 6**.



..... mol of HCl were present in the graduated flask.

- (iii) Calculate how many moles of hydrochloric acid were present in 250 cm^3 of **FA 4**.

250 cm^3 of **FA 4** contained mol HCl.

- (iv) Calculate the following.

(answer to (d)(iii) – answer to (d)(ii))

This is the amount of hydrochloric acid that reacted with the calcium carbonate in the weighed sample of **FA 3**.

..... mol of HCl reacted with the calcium carbonate in g **FA 3**.

- (v) Use your answer to (iv) to calculate the mass of calcium carbonate that reacted with hydrochloric acid.
This is the mass of pure CaCO_3 in the weighed sample of **FA 3**.



[A_r : Ca, 40.0; C, 12.0; O, 16.0]

The weighed sample of **FA 3** contains g of CaCO_3 .

- (vi) Calculate the percentage of calcium carbonate, CaCO_3 , in **FA 3** by evaluating the following expression.

$$\frac{\text{mass of } \text{CaCO}_3 \text{ from (d)(v)}}{\text{mass of FA 3 used, from (a)}} \times 100$$

Complete your evaluation even if your answer is greater than 100%

FA 3 contains % calcium carbonate.
[5]

- (e) 6.25 g of pure calcium carbonate are required to neutralise all the hydrochloric acid in 250 cm^3 of **FA 4**.

You were instructed to measure a mass between 2.60 g and 2.80 g of **FA 3** in this experiment.

What difficulties might you encounter if you used a mass of about 5.50 g of **FA 3** in this experiment?

.....
.....
.....

[1]

(f) (i) Complete the following table.

The balance used in the experiment displays the mass to	decimal places.
The maximum error in a single balance reading is ±	g.
The maximum error in measuring the mass of FA 3 is ±	g.

(ii) Calculate the maximum percentage error in the mass of FA 3 measured in (a).

The maximum error in the mass of FA 3 is %.
[2]

(g) (i) The percentage of calcium carbonate in the weighed sample of FA 3 can also be found by investigating the thermal decomposition of the compound into calcium oxide and carbon dioxide.

Write a balanced equation, including state symbols, for this thermal decomposition.

(ii) **Briefly outline** the key measurements to be made in order to find the percentage of calcium carbonate in FA 3 by this method.

1.
2.
3.
4.
5.
6.

(You do not have to use all of the numbered steps in your answer)

[2]

[Total: 14]

- 3 **FA 7, FA 8** and **FA 9** are aqueous solutions, each containing one cation and one anion from those listed on pages 13 and 14 in the Qualitative Analysis Notes.

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling-tube MUST be used.

Rinse and reuse test-tubes wherever possible.

- (a) Use aqueous sodium hydroxide and aqueous ammonia, in separate tests, to identify the cation present in **FA 7, FA 8** and **FA 9**.

Present your results for each of the solutions in a suitable form below.

Conclusion

Complete the following table.

<i>solution</i>	<i>cation</i>	<i>supporting evidence</i>
FA 7		
FA 8		
FA 9		

- (b) (i) **FA 7, FA 8 and FA 9** each contain a single anion which may be Cl^- , I^- or SO_4^{2-} .

Suggest a reagent that would enable you to identify any solutions containing SO_4^{2-} .

Reagent

Use this reagent to test each of the solutions. Record your observations in the table below. Indicate, with a tick in the final column, any solution containing SO_4^{2-} .

<i>solution</i>	<i>observation</i>	SO_4^{2-} present
FA 7		
FA 8		
FA 9		

- (ii) Select a further reagent that will enable you to identify the halide ion present in any remaining solution(s).

Reagent

Use this reagent to test the remaining solution(s).

Record your observations and the identity of the halide in a suitable form in the space below.

- (c) **FA 10** is a white crystalline solid which turns into another white solid, **FA 11**, when heated strongly.

Carry out the tests on **FA 10** and **FA 11** in the table below.

Observe carefully at each stage and record all of your observations in the table.

	<i>test</i>	<i>observations</i>
(i)	Place 1 spatula measure of FA 10 in a hard glass test-tube. Heat the solid very strongly until no further change is seen.	
(ii)	Place 1 small spatula measure of FA 11 in a test-tube and add 1 cm depth of dilute hydrochloric acid.	
As soon as you have completed your observation in (ii), fill the test-tube with water.		

[5]

[Total: 16]

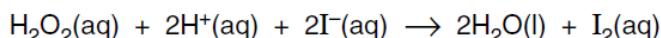
Practical 9

1 Read through question 1 before starting any practical work.

You are provided with the following reagents.

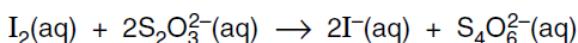
- FB 1**, 1 mol dm⁻³ sulphuric acid, H₂SO₄
- FB 2**, 0.1 mol dm⁻³ potassium iodide, KI
- FB 3**, 0.1 mol dm⁻³ sodium thiosulphate, Na₂S₂O₃
- FB 4**, 0.1 mol dm⁻³ hydrogen peroxide, H₂O₂
- starch solution
- distilled water

In the presence of an acid, iodide ions are oxidised by hydrogen peroxide to iodine.



The rate of reaction can be followed by timing the formation of a fixed amount of iodine in the solution.

If sodium thiosulphate is present in the reaction mixture it reacts with the iodine formed and the solution remains colourless.

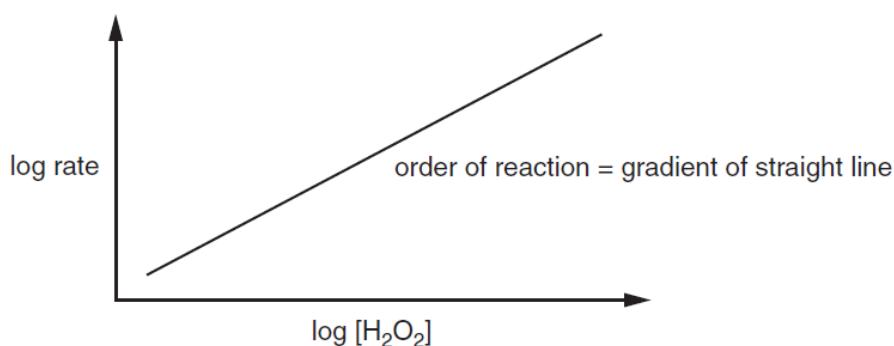


When all of the sodium thiosulphate present has reacted, iodine, I₂, will appear in the solution producing an immediate blue colour with starch indicator.

In a series of experiments where the concentration of a reagent is changed

- 1/time can be used as a measure of rate,
- the volume of the reagent used can be taken as a measure of its concentration providing the total volume of the mixture is kept constant in each experiment.

The order of reaction with respect to hydrogen peroxide can be obtained by plotting a graph of log rate against log [H₂O₂].



(a) Method

Experiment 1

- Fill the burette **labelled FB 3** with **FB 3** and the burette **labelled FB 4** with **FB 4**.
- Use the measuring cylinder **labelled A** to put the following solutions into a 250 cm^3 conical flask:
 - 20 cm^3 of **FB 2**, potassium iodide,
 - 20 cm^3 of distilled water.
- Add to the flask from the burette 1.00 cm^3 of **FB 3**, sodium thiosulphate.
- Add six drops of starch indicator to the mixture in the flask.
- Run 20.00 cm^3 of **FB 4**, hydrogen peroxide, from the second burette into a 100 cm^3 beaker.
- Use the measuring cylinder **labelled B** to add 20 cm^3 of **FB 1**, sulphuric acid, to the hydrogen peroxide in the beaker.
- Tip the contents of the beaker into the conical flask and **immediately** start a stop-clock or note the start time on a clock with a second hand.
- Swirl the flask to mix the reagents.
- Observe the solution and stop the clock or note the time when the solution suddenly turns blue.
- Record the time taken to the nearest second.

The time taken is s.

Experiment 2

- Empty, thoroughly rinse and drain the conical flask used in experiment 1.
- Use the measuring cylinder **labelled A** to put the following solutions into the 250 cm^3 conical flask:
 - 20 cm^3 of **FB 2**, potassium iodide,
 - 35 cm^3 of distilled water.
- Add to the flask from the burette 1.00 cm^3 of **FB 3**, sodium thiosulphate.
- Add six drops of starch indicator to the mixture in the flask.
- Run 5.00 cm^3 of **FB 4**, hydrogen peroxide, from the second burette into a 100 cm^3 beaker.
- Use the measuring cylinder **labelled B** to add 20 cm^3 of **FB 1**, sulphuric acid, to the hydrogen peroxide in the beaker.
- Tip the contents of the beaker into the conical flask and **immediately** start a stop-clock or note the start time on a clock with a second hand.
- Swirl the flask to mix the reagents.
- Observe the solution and again stop the clock or note the time when the solution suddenly turns blue.
- Record the time taken to the nearest second.

The time taken is s.

[2]

- (b) In experiment 1 you will have obtained the time taken for a 'fast' reaction and in experiment 2 the time taken for a 'slow' reaction.

You are to repeat the experiment with further mixtures in which **only** the concentration of hydrogen peroxide is varied.

In selecting the mixtures to be used you should consider

- how many mixtures need to be used,
- what concentrations of hydrogen peroxide should be used,
- what range these concentrations should cover,
- that only the concentration of hydrogen peroxide must change.

Remember – you already have reaction times for two mixtures with different concentrations of hydrogen peroxide.

In the space below prepare to record, in an appropriate form, the results of the experiments you will perform and the results of experiments 1 and 2.

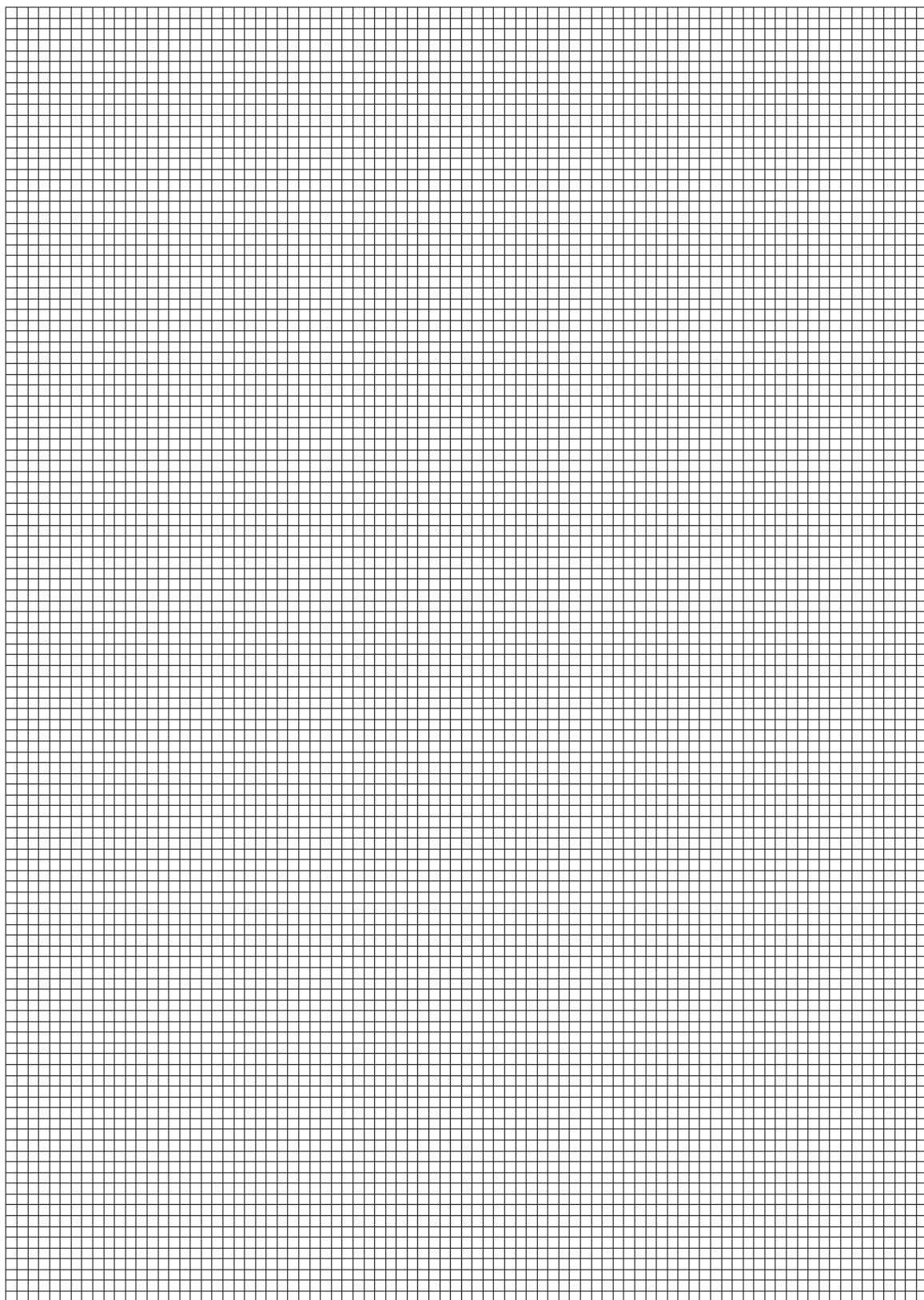
Your recorded results should include calculated values to enable you to plot

log (1/time) against **log (volume of FB 4)**.

Carry out the additional experiments and record your results.

[11]

- (c) Use the grid below to plot a graph of **log (1/time)** against **log (volume of FB 4)**.
Draw an appropriate straight line through the points plotted.



[4]

- (d) Draw construction lines on your graph and obtain data from them to enable you to calculate the gradient of the graph.

Calculate the gradient of the line drawn, which is the order of reaction with respect to hydrogen peroxide.

The reaction is order with respect to hydrogen peroxide, H_2O_2 . [3]

- (e) In experiment 1 burettes and measuring cylinders were used to measure volume.

A burette is graduated to 0.10 cm^3 and is usually read to the nearest 0.05 cm^3 .

A 25 cm^3 measuring cylinder is graduated to 0.5 cm^3 .

Estimate the error when measuring a volume of 20 cm^3 in a 25 cm^3 measuring cylinder.

The error is \pm cm^3 .

Use this answer and the information above to calculate the percentage error for each volume measurement made in experiment 1. Complete the table below.

solution	apparatus used	volume / cm^3	error / cm^3	% error
FB 1, FB 2, distilled water	25 cm^3 measuring cylinder	20		
FB 3	burette	1.00		
FB 4	burette	20.00		

Identify the most significant source of error in this experiment.

..... [3]

- (f) The experimental method can be modified to enable the rate of reaction with respect to iodide ions, I^- , to be investigated.

You will perform two further experiments **using the method for experiment 1 in section (a)**. You will keep the concentration of hydrogen peroxide constant and **reduce** the concentration of iodide ions.

First copy your reaction time from experiment 1 in section (a) into the table below. Then complete the table below to show the volumes of **FB 2** and distilled water you will use in these two further experiments.

Carry out each experiment as before and record the time taken in each case.

	volume FB 1 (H_2SO_4) /cm ³	volume FB 2 (KI) /cm ³	volume water /cm ³	volume FB 3 ($Na_2S_2O_3$) /cm ³	volume FB 4 (H_2O_2) /cm ³	reaction time /s
experiment 1 in section (a)	20	20	20	1.00	20.00	

experiment i	20			1.00	20.00	
experiment ii	20			1.00	20.00	

[1]

- (g) Use the experimental results from the three experiments to draw a conclusion as to how the rate of reaction is affected by changing the concentration of iodide ions.

.....
.....
.....

[1]

[Total: 25]

- 2 The three solutions **FB 5**, **FB 6**, and **FB 7 each** contain **one** of the following.

copper(II) chloride, CuCl_2
chromium(III) chloride, CrCl_3
nickel(II) sulphate, NiSO_4

- (a) Use information from the Qualitative Analysis Notes on page 12 to select a pair of reagents that you could use to determine which solution contains the sulphate ion.

Carry out the tests and record, in the space below, the reagents used and the observations made.

From these tests, solution **FB** contains the sulphate ion.

[3]

- (b) The solutions containing copper(II) and chromium(III) ions can be distinguished from one another by adding either aqueous sodium hydroxide or aqueous ammonia. Nickel(II) ions behave in a similar way to copper(II) ions with these reagents.

Add NaOH(aq) and $\text{NH}_3\text{(aq)}$ separately to each of the solutions **FB 5**, **FB 6** and **FB 7**.

Record your observations in the space below.

From your observations in **(a)** and **(b)** identify the solutions containing copper(II) ions and chromium(III) ions.

Solution contains Cu^{2+} .

supporting evidence

.....

Solution contains Cr^{3+} .

supporting evidence

.....

[5]

- (c)** You are to perform the tests given in the table below on the solid **FB 8** and to comment on the type of compound present in **FB 8**.

Record details of colour changes seen, the formation of any precipitate and the solubility of any such precipitate in an excess of the reagent added.

Where gases are released they should be identified by a test, **described in the appropriate place in your table**.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests should be attempted.

test	observations with FB 8
<p>(i) To 2 cm depth of distilled water in a boiling-tube, add 1 spatula measure of FB 8.</p> <p>Warm to dissolve the solid and immediately add a 1 cm length of magnesium ribbon.</p>	
<p>(ii) To 1 cm depth of aqueous sodium hydroxide in a test-tube, add 1 spatula measure of FB 8.</p> <p>Stir the mixture, then add 2 cm depth of dilute hydrochloric acid.</p> <p>Stir the mixture, then add 3 cm depth of aqueous sodium hydroxide.</p>	

test	observations with FB 8
<p>(iii) To 1 cm depth of ethanol in a boiling-tube, add 2 spatula measures of FB 8 and a few drops of concentrated sulphuric acid (CARE: corrosive). Heat the contents of the tube for 1-2 minutes, <i>using the apparatus provided for heating a flammable liquid (CARE)</i>. Pour the contents of the tube into a 100 cm³ beaker full of water.</p>	

From your observations, draw conclusions about the type of compound present in **FB 8**.

.....

.....

.....

[7]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate.]

1 Reactions of aqueous cations

	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)	no ppt. 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
lead(II), Pb ²⁺ (aq)	white ppt. soluble in excess	white 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

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow soln turns orange with H^+ (aq); gives yellow ppt. with Ba^{2+} (aq); gives bright yellow ppt. with Pb^{2+} (aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+ (aq) (soluble in NH_3 (aq)); gives white ppt. with Pb^{2+} (aq)
bromide, Br^- (aq)	gives cream ppt. with Ag^+ (aq) (partially soluble in NH_3 (aq)); gives white ppt. with Pb^{2+} (aq)
iodide, I^- (aq)	gives yellow ppt. with Ag^+ (aq) (insoluble in NH_3 (aq)); gives yellow ppt. with Pb^{2+} (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 acid); gives white ppt. with Pb^{2+} (aq)
sulfite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+} (aq) (soluble in excess dilute strong acid)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
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 acidified aqueous potassium dichromate(VI) from orange to green

INSERT

2 hours