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9701/31

May/June 2008

2 hours

Additional Materials: As listed in the Instructions to Supervisors

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use a soft pencil for any diagrams, graphs or rough working.
Do **not** use staples, paper clips, highlighters, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
You are advised to show all working in calculations.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
Total	

This document consists of **11** printed pages and **1** blank page.

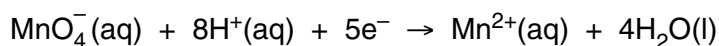
1 Read through question 1 before starting any practical work.

For
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Use

You are provided with the following reagents.

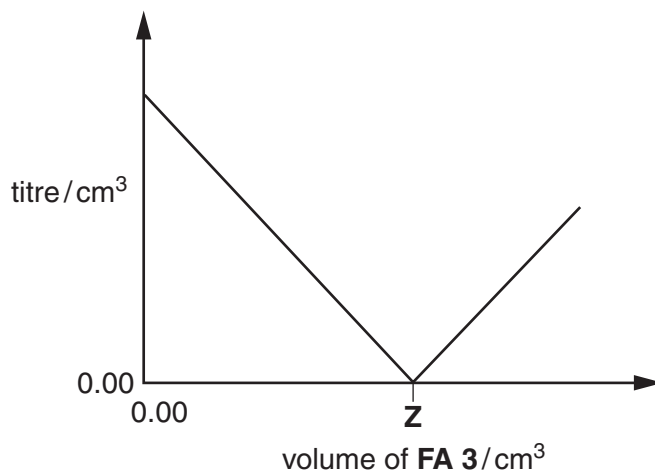
- **FA 1** containing 15.68 g dm^{-3} of hydrated ammonium iron(II) sulphate $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$
- **FA 2**, $0.015 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4
- **FA 3** containing $0.025 \text{ mol dm}^{-3}$ of a reagent **X**
- 1.0 mol dm^{-3} sulphuric acid, H_2SO_4

Iron(II) ions, Fe^{2+} , are oxidised by acidified manganate(VII) ions.



Reagent **X** oxidises Fe^{2+} to Fe^{3+} and is also oxidised by acidified MnO_4^- .

If varying volumes of **FA 3**, containing reagent **X**, are added to 25.0 cm^3 of **FA 1** in the presence of H_2SO_4 and the mixtures are titrated against **FA 2**, a graph of the results can be drawn as shown.



You are to determine experimentally

- **Z**, the exact volume of **FA 3** which reacts with 25.0 cm^3 of **FA 1**,
- the mole ratio for the reaction of **FA 1** with reagent **X**.

(a) Method

- Fill a burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Use a measuring cylinder to add approximately 10 cm^3 of 1.0 mol dm^{-3} H_2SO_4 to the solution in the flask.
- Titrate the **FA 1** in the flask with **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately 13 cm^3 of **FA 2**.

One titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

For
Examiner's
Use

[1]

(b) Method

- Fill the second burette with **FA 3**.
- Empty and rinse the conical flask used in part **(a)**.
- Pipette 25.0 cm^3 of **FA 1** into the conical flask and add 10 cm^3 of H_2SO_4 using a measuring cylinder.
- Run 12.00 cm^3 of **FA 3** from the second burette into the flask.
- Titrate against **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately 5 cm^3 of **FA 2**.

One titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

[1]

- (c)** The volume of **FA 3** added to the flask in **(b)** reacts with some but not all of the **FA 1** present. Calculate the difference between the titres obtained in parts **(a)** and **(b)**. Use this difference and the volume of **FA 3** added to the flask in **(b)** to calculate the volume of **FA 3** that you would expect to react with all of the Fe^{2+} ions in 25.0 cm^3 of **FA 1**.

volume of **FA 3** = ... cm^3
[1]

- (d) The value you have obtained in (c) is an **approximate** value of **Z**. You are to perform four more titrations, **each** with a **different** volume of **FA 3** added to 25.0 cm³ of **FA 1**, in order to plot a graph of the form shown on page 2 and to obtain an exact value for **Z**.

One titration, performed accurately, will be sufficient for each volume of **FA 3** added.

The volume of **FA 3** you have obtained in (c) will help you to choose suitable volumes of **FA 3** to be added for each titration.

(If you were unable to calculate the volume of **FA 3** in (c) assume that the value lies in the range 19.0 cm³ to 21.0 cm³.)

Remember

- you should not use more than 40.0 cm³ of **FA 3** for any single titration,
- you already have data for titrations with no **FA 3** added and with 12.00 cm³ added.

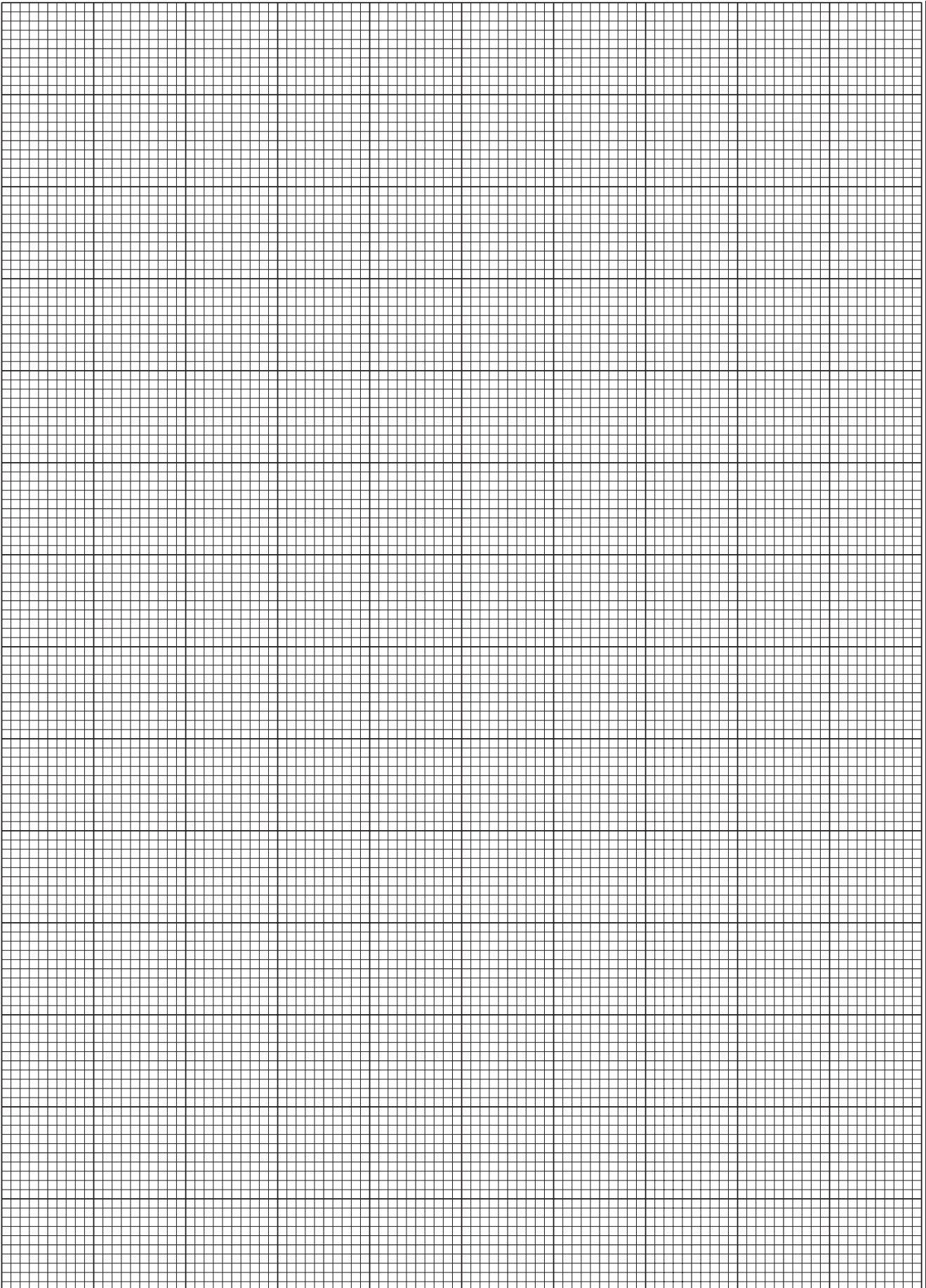
Prepare a table in the space below and use it to record the titration results for each volume of **FA 3** added. Include in your table the titre values from parts (a) and (b).

[6]

- (e) Use the grid on the opposite page to plot a graph of titre against volume of **FA 3** added.

Draw **two** straight lines through the plotted points to find **Z**, the volume of **FA 3** that just reacts with the Fe²⁺ ions in 25.0 cm³ of **FA 1**.

Z, the volume of **FA 3** read from the graph is cm³.



- (f) Circle on the graph **one** point where a repeat titration might be appropriate and justify your decision. If you do not think that any titration needs to be repeated, explain why you have come to that conclusion.

For
Examiner's
Use

.....

.....

.....

[1]

Calculations

Show your working and appropriate significant figures in **all** of your calculations. [2]

- (g) Calculate how many moles of Fe^{2+} ions were pipetted into the flask.
[A_r : Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

..... mol of Fe^{2+} were pipetted into the flask. [2]

- (h) Calculate how many moles of **X** are present in **Z**, the volume of **FA 3** read from your graph.

..... mol of **X** were present in cm^3 of **FA 3**. [1]

- (i) Calculate, to 3 significant figures, the number of moles of Fe^{2+} ions that react with 1 mol of **X**.

[2]

[Total: 24]

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- 2 The three solutions **FA 4**, **FA 5**, and **FA 6** each contain **one** of the following.
- aluminium sulphate, $Al_2(SO_4)_3$
 - ammonium iodide, NH_4I
 - zinc nitrate, $Zn(NO_3)_2$
- (a) Use the information on page 12 to select two suitable reagents to use to discover which solution contains iodide ions.

Record, in the space below, the reagents used and the observations made.

From these tests, solution **FA** contains iodide ions. [5]

You are to perform the tests given in the table opposite on each of **FA 4**, **FA 5** and **FA 6** to identify, where possible, the cation and anion present in each solution.

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 for ions present should be attempted.

test	observations with FA 4	observations with FA 5	observations with FA 6
(b) To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide drop-by-drop until it is in excess.			
(c) To 1 cm depth of solution in a test-tube, add aqueous ammonia drop-by-drop until it is in excess.			
(d) To 1 cm depth of solution in a test-tube, add aqueous barium chloride, then			
add dilute hydrochloric acid.			
(e) To 1 cm depth of solution in a boiling-tube add 2 cm depth of water and 1 cm depth of aqueous lead(II) nitrate, then			
if a precipitate has formed, cautiously warm until the solution boils, then			
cool the tube by standing it in a beaker of cold water.			

[6]

- (f) For each of the solutions **FA 4**, **FA 5**, and **FA 6**, summarise the evidence from the tests performed to identify the cations and anions present. State clearly where a cation or an anion has **not** been specifically identified.

FA 4 contains

supporting evidence

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

FA 5 contains

supporting evidence

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

FA 6 contains

supporting evidence

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

[4]

- (g) When testing a solution containing both NH_4I and $\text{Zn}(\text{NO}_3)_2$, suggest why a student should identify the NH_4^+ ion before attempting to identify the NO_3^- ion. The Qualitative Analysis Notes on pages 11 and 12 should help you to answer this.

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

[Total: 16]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	ammonia produced on heating	
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. 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

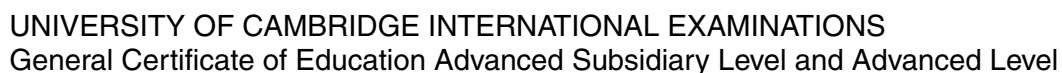
<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow solution turns orange with H^+ (aq); gives yellow ppt. with Ba^{2+} (aq); gives bright yellow ppt. with Pb^{2+} (aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+ (aq) (soluble in NH_3 (aq)); gives white ppt. with Pb^{2+} (aq)
bromide, Br^- (aq)	gives pale 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 $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+} (aq) or with Pb^{2+} (aq) (insoluble in excess dilute strong acids)
sulphite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+} (aq) (soluble in excess dilute strong acids)

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
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green

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9701/32

May/June 2008

2 hours

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READ THESE INSTRUCTIONS FIRST

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Session
Laboratory

For Examiner's Use	
1	
2	
Total	

This document consists of **12** printed pages.

- 1 You are provided with the following.

FB 1, 3.00 mol dm^{-3} hydrochloric acid, HCl

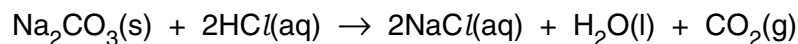
Three tubes containing different mixtures of sodium carbonate, Na_2CO_3 , and sodium hydrogencarbonate, NaHCO_3 , each with a total mass of 5.00 g of mixture.

For
Examiner's
Use

tube labelled	mass of Na_2CO_3 /g	mass of NaHCO_3 /g	% by mass of Na_2CO_3
FB 2	1.00	4.00	20.0
FB 3	2.50	2.50	50.0
FB 4	4.00	1.00	80.0

You are to determine the temperature change, ΔT , when the contents of each of the tubes **FB 2**, **FB 3** and **FB 4** react with an excess of hydrochloric acid, **FB 1**.

- (a) (i) Calculate the volume of 3.00 mol dm^{-3} hydrochloric acid required to react with 5.00 g of sodium carbonate, Na_2CO_3 . Show your working.



$[M_r: \text{Na}_2\text{CO}_3, 106.0]$

- (ii) Calculate the volume of 3.00 mol dm^{-3} hydrochloric acid required to react with 5.00 g of sodium hydrogencarbonate, NaHCO_3 . Show your working.



$[M_r: \text{NaHCO}_3, 84.0]$

[2]

35.00 cm³ of 3.00 mol dm⁻³ HCl will be used in each experiment – an excess of hydrochloric acid.

For
Examiner's
Use

(b) Read the following instructions before starting this section.

- Fill a burette with **FB 1**, 3.00 mol dm⁻³ HCl.
- Support the plastic cup in a 250 cm³ beaker.
- Run 35.00 cm³ of acid from the burette into the cup.
- Measure the steady temperature of the acid in the plastic cup.
- Tip the contents of the tube labelled **FB 2** into the acid as quickly as possible but take care to avoid overflow or acid spray. Stir with the thermometer, measure and record the highest **or** lowest temperature reached in the reaction.
- Make certain that all of the solid has been transferred from the tube to the cup. Tap the tube if necessary to dislodge any residual solid.
- Empty and rinse the plastic cup with water. Shake out any residual drops of water.
- Repeat the experiment for each of the tubes **FB 3** and **FB 4**.

Record all measurements of temperature and the temperature changes, ΔT , in an appropriate form in the space below. Indicate clearly whether the temperature has increased or decreased in the reaction.

[4]

- (c)** Select masses of Na₂CO₃ and NaHCO₃ which can be used to prepare two further tubes, each containing a mixture which can be used in the same experiment as described above. The temperature change when each of these mixtures reacts with hydrochloric acid will be used with those above to plot five points on a graph.

tube labelled	mass of Na ₂ CO ₃ /g	mass of NaHCO ₃ /g
FB 5		
FB 6		

[1]

(d) Preparation of the tubes FB 5 and FB 6

You are provided with

- empty tubes labelled **FB 5** and **FB 6**,
- sodium carbonate and sodium hydrogencarbonate.

Prepare each tube and record in an appropriate form in the space below

- all of your weighings,
- the mass of sodium carbonate in the mixture,
- the mass of sodium hydrogencarbonate in the mixture,
- the % by mass of sodium carbonate in the mixture.

[4]

(e) Carry out the same experiment as in (b) for each of the tubes FB 5 and FB 6.

Record all temperature readings and the temperature change, ΔT , for each of the tubes.

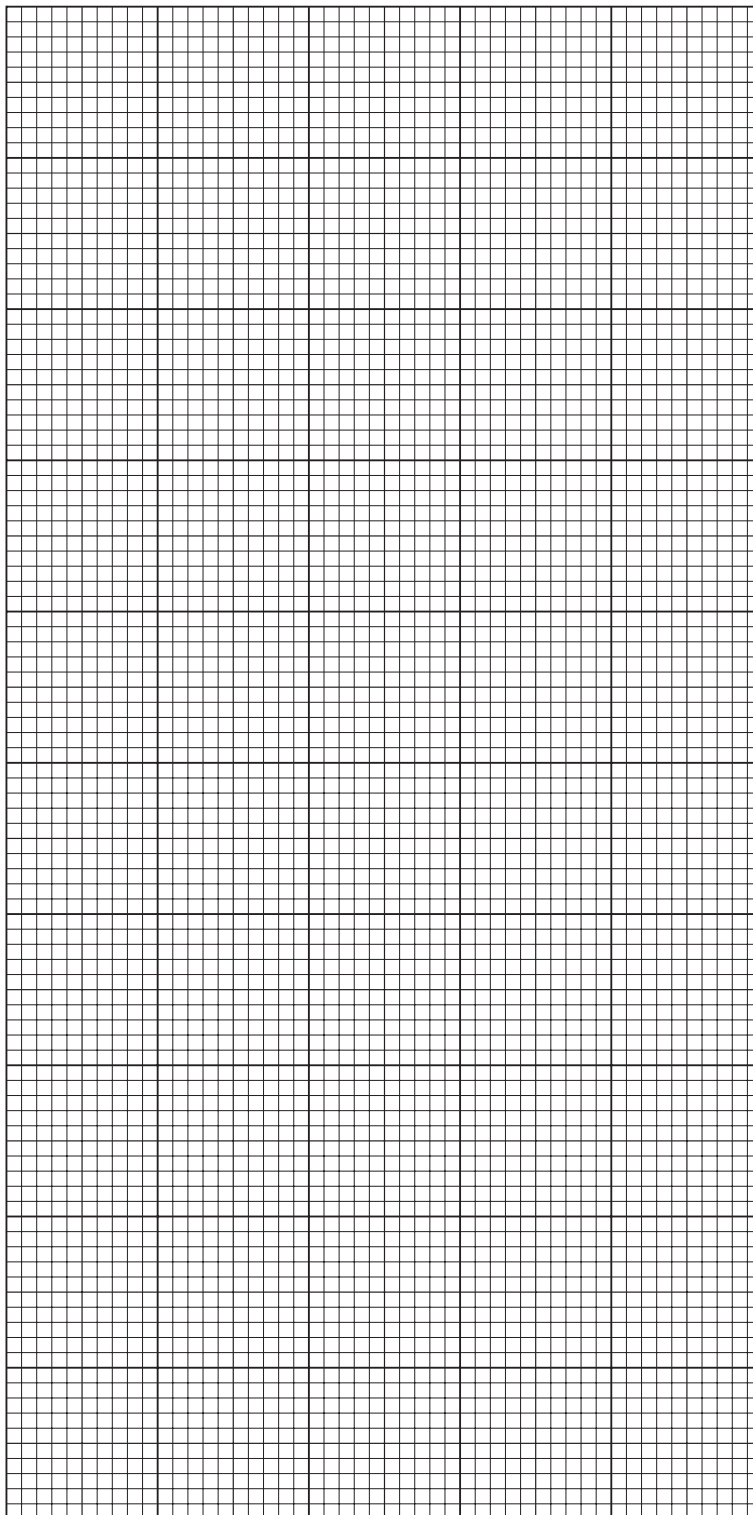
[2]

- (f) Plot ΔT (y-axis, starting at -10°C) against the % by mass of Na_2CO_3 in the mixture (x-axis, starting at 0%).

Remember – the temperature has increased in some experiments and decreased in others.

Draw the most appropriate straight line through the five plotted points. Extend this line until it crosses the y-axis.

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Use



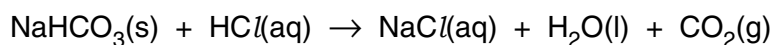
[4]

- (g) Record from the graph the temperature change when the mixture contains no sodium carbonate. This represents the temperature change for 5.00 g of sodium hydrogencarbonate.

ΔT , read from the graph for 0% sodium carbonate is $^{\circ}\text{C}$. [1]

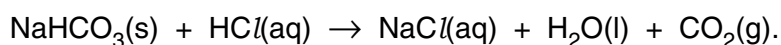
- (h) You are to use the value in (g), obtained from the graph, to calculate the enthalpy change for the reaction between sodium hydrogencarbonate and hydrochloric acid.

For
Examiner's
Use



- (i) Calculate the energy change in the plastic cup when 5.00 g of NaHCO_3 reacts with excess hydrochloric acid.
[4.3 J are absorbed or released when the temperature of 1.0 cm^3 of solution changes by 1°C .]

- (ii) Calculate the enthalpy change, ΔH , for the reaction



Give your answer in kJ mol^{-1} , correct to **3 significant figures**.
Include the appropriate sign.

[M_r : NaHCO_3 , 84.0]

$\Delta H = \dots\dots\dots \text{kJ mol}^{-1}$
[4]

- (i) Suggest the most significant source of error in this experiment.

.....

.....

..... [1]

- (j) Suggest a modification to the experimental procedure that would reduce the error described in (i).

.....

.....

.....

.....

..... [1]

- (k) Do you think the method used is capable of producing an accurate value for the enthalpy change for the reaction of sodium hydrogencarbonate and hydrochloric acid?

Justify your answer by referring to the results of your experiment.

.....

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.....

.....

..... [1]

[Total: 25]

2 The three solutions **FB 7**, **FB 8**, and **FB 9** each contain one of the following.

a cation and the chloride ion
manganese(II) sulphate, MnSO_4
magnesium sulphate, MgSO_4

- (a) Using the information on page 12 select **two** suitable reagents and use them to carry out a test to determine which solutions contain the sulphate ion.

In the space below, record details of the test performed and the observations made.

From this test, solutions **FB** and **FB** contain the sulphate ion.

[2]

- (b) By selecting a further **two** reagents, carry out a test to confirm the presence of the chloride ion in the remaining solution.

In the space below, record details of the test performed and the observations made.

[2]

You are to perform the tests given in the table opposite on each of **FB 7**, **FB 8** and **FB 9** to identify and confirm the cation present in the solution.

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 an appropriate place in the table.

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.

test	observations with FB 7	observations with FB 8	observations with FB 9
(c) To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide drop-by-drop until it is in excess.			
(d) To 1 cm depth of solution in a test-tube, add aqueous ammonia drop-by-drop until it is in excess.			

[4]

- (e)** Observations made with aqueous sodium hydroxide should have indicated that one of the solutions contains either of two cations. Identify this solution and the **two** possible cations.

Solution could contain or

Make use of the Qualitative Analysis Notes on page 11 to suggest what further test you could do to identify which of the two ions was present.

.....

Carry out your suggestion using a boiling-tube. Record the results below and explain how this enables you to identify the ion present.

.....

[3]

- (f) For each of the solutions **FB 7**, **FB 8**, and **FB 9**, identify the cation present and give supporting evidence from the observations made.

FB 7 contains

supporting evidence

.....

.....

.....

FB 8 contains

supporting evidence

.....

.....

.....

FB 9 contains

supporting evidence

.....

.....

.....

[3]

- (g) Carry out the tests below on **FB 7**.

test	observations
To 1 cm depth of solution in a boiling-tube, add 2 cm depth of aqueous ammonia, then slowly add 2 cm depth of aqueous hydrogen peroxide.	
To 1 cm depth of solution in a boiling-tube, add 2 cm depth of aqueous hydrogen peroxide, then slowly add 2 cm depth of aqueous ammonia.	

[1]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH ₄ ⁺ (aq)	ammonia produced on heating	
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.
chromium(III), Cr ³⁺ (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe ²⁺ (aq)	green ppt. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. 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 solution 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 pale 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 $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+} (aq) or with Pb^{2+} (aq) (insoluble in excess dilute strong acids)
sulphite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+} (aq) (soluble in excess dilute strong acids)

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
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green