



UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

CANDIDATE
NAME

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CENTRE
NUMBER

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NUMBER

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CHEMISTRY

9701/31

Advanced Practical Skills 1

May/June 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 12 and 13.

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	

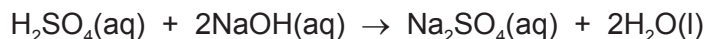
For Examiner's Use	
1	
2	
3	
Total	

This document consists of **12** printed pages and **4** blank pages.



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- 1 The reaction between sulfuric acid and sodium hydroxide is exothermic.



By measuring the temperature changes that occur when different volumes of the acid are added to a fixed volume of the alkali, it is possible to determine the neutralisation point. This is the point at which just enough acid has been added to react with all the alkali present. The aim of the investigation is to determine the concentration of the sulfuric acid.

FA 1 is 2.00 mol dm^{-3} sodium hydroxide, NaOH .

FA 2 is dilute sulfuric acid, H_2SO_4 .

Read through the instructions carefully and prepare a table for your results before starting any practical work.

(a) Method

- Support a plastic cup in a 250 cm^3 beaker.
- Use a pipette to transfer 25.0 cm^3 of **FA 1** into the plastic cup.
- Record the temperature of **FA 1**, T_1 , in the space below.

$T_1 = \dots\dots\dots ^\circ\text{C}$

- Fill the burette labelled **FA 2** with **FA 2**.
- Add 5.00 cm^3 of **FA 2** from the burette to the plastic cup.
- Stir the mixture thoroughly and record the temperature of the solution.
- Add a further 5.00 cm^3 of **FA 2** to the plastic cup and again record the temperature.
- Repeat the addition of 5.00 cm^3 portions of **FA 2** until you have added a total of 50.00 cm^3 of **FA 2** to the plastic cup. Measure the temperature after each addition.
- Record in your table below the total volume of **FA 2** added and the temperature of the solution after each addition.

For
Examiner's
Use

I	
II	
III	
IV	
V	

[5]

$$\Delta T = \text{temperature recorded} - T_1.$$

V_T = volume of **FA 2** + volume of **FA 1**.

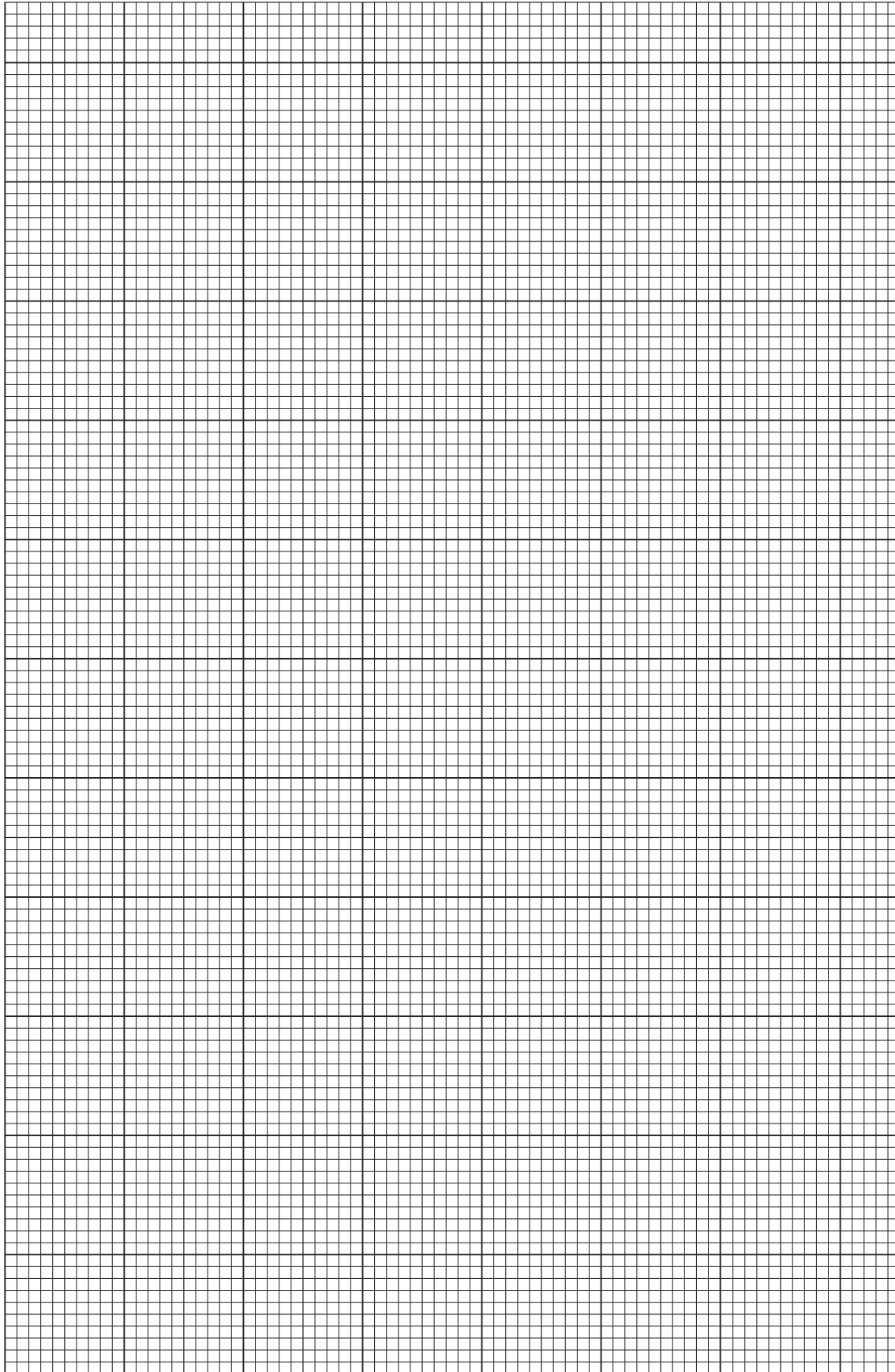
Use your experimental results to complete the following table. You should include:

- the volume of **FA 2**
- the total volume in the plastic cup, V_T
- the temperature of the solution
- the temperature rise, ΔT
- the total volume $\times$ the temperature rise, $(V_T \times \Delta T)$

[1]

- (c) (i) On the grid below, plot the values of $(V_T \times \Delta T)$ on the y-axis against the volume of **FA 2** on the x-axis.

For
Examiner's
Use



I	
II	
III	
IV	

- (ii) Draw a straight line of best fit through the points where the values of $(V_T \times \Delta T)$ are increasing. Draw a second straight line of best fit through the points where the values of $(V_T \times \Delta T)$ are decreasing.

- (iii) From your graph, determine the volume of **FA 2** where the two lines of best fit intersect.

volume of **FA 2** = ... cm^3
[5]

- (d) The value you recorded in (c)(iii) is the volume of **FA 2** which is needed to neutralise 25.0 cm^3 of **FA 1**. In the following calculations you will determine the concentration of **FA 2**.

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

- (i) Calculate how many moles of sodium hydroxide are contained in 25.0 cm^3 of **FA 1**.

moles of NaOH = ... mol

- (ii) Calculate how many moles of sulfuric acid would react with the number of moles of NaOH in (i).

moles of H_2SO_4 = ... mol

- (iii) Calculate the concentration of **FA 2**.

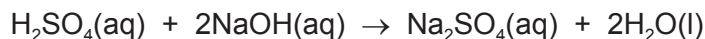
concentration of **FA 2** = ... mol dm^{-3}
[3]

- (e) Other than heat losses from the plastic cup to the surroundings, suggest an additional source of error in this experiment and how this error could be reduced.

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

[Total: 15]

- 2 A second way to determine the concentration of an acid is by volumetric titration. In this experiment you will first dilute the sample of **FA 2** that you used in **Question 1** and then titrate this diluted solution using aqueous sodium hydroxide.



FA 2 is dilute sulfuric acid, H_2SO_4 .

FA 3 is $0.150 \text{ mol dm}^{-3}$ sodium hydroxide, NaOH .
distilled water

(a) Method

Dilution of FA 2

- Use the burette labelled **FA 2** to transfer 25.00 cm^3 of **FA 2** into the 250 cm^3 graduated (volumetric) flask, labelled **FA 4**.
- Make up the contents of the flask to the 250 cm^3 mark with distilled water.
- Stopper the flask and mix the contents thoroughly. This is solution **FA 4**.

Titration

- Fill the burette labelled **FA 3** with **FA 3**.
- Use a clean pipette to transfer 25.0 cm^3 of **FA 4** into a conical flask.
- Add to the flask a few drops of the acid-base indicator provided.
- Titrate the acid in the flask with the alkali, **FA 3**.

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 3** added in each accurate titration. Make certain that any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	

[5]

- (b) 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 4** required cm³ of **FA 3**.
[1]

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

moles of NaOH = ... mol

- (ii) Hence, calculate how many moles of H₂SO₄ are contained in 25.0 cm³ of **FA 4**.

moles of H₂SO₄ = ... mol

- (iii) Calculate the concentration of the sulfuric acid, **FA 2**.

concentration of **FA 2** = ... mol dm⁻³
[3]

- (d) You have used two methods to determine the concentration of the sulfuric acid in **FA 2**. Use your answers to **1(d)(iii)** and **2(c)(iii)** to calculate the difference in these values as a percentage of the value found by the volumetric titration method.

percentage difference = ... %
[1]

[Total: 10]

I	
II	
III	

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.

(a) **FA 5, FA 6, FA 7** and **FA 8** are aqueous solutions each of which contains a single cation and a single anion. Some of the ions present are listed below.



By observing the reactions that occur when pairs of the solutions are mixed together, you will be able to identify which solution contains which of these ions.

Use a 1 cm depth of each solution in a test-tube and record your observations in the following table.

For
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Use

	FA 6	FA 7	FA 8
FA 5			
FA 6			
FA 7			

[8]

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(b) From your observations deduce which solution contains each of the following ions.

ion	Pb^{2+}	Cl^-	CO_3^{2-}	CrO_4^{2-}
solution				

[2]

I	
II	

(c) Identify another ion that is present in one of the solutions. Explain your reasoning.

ion

explanation

..... [1]

- (d) (i) If chloride ions, Cl^- , were to be replaced with bromide ions, Br^- , in one of the solutions, would it make any difference to the observations you made in (a)? Explain your answer.

.....

.....

.....

- (ii) **FA 9** is an aqueous solution containing either chloride ions or bromide ions. Select a pair of reagents to identify which anion is present.

reagents

Carry out this test and record your observations and conclusion.

observations

.....

The anion in **FA 9** is

[4]

[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)	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), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

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NUMBER

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CHEMISTRY

9701/32

Advanced Practical Skills 2

May/June 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
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Do not use staples, paper clips, highlighters, glue or correction fluid.
DO **NOT** WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 10 and 11.

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

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1	
2	
Total	

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

- 1 You will determine the enthalpy change, ΔH , for the reaction of sodium carbonate, Na_2CO_3 , with water and carbon dioxide to form sodium hydrogencarbonate, NaHCO_3 . In step 1 you will react a known mass of sodium hydrogencarbonate with a known volume of dilute hydrochloric acid and find the temperature change. In step 2 you will find the temperature change on adding a known mass of sodium carbonate to a known volume of acid. You will then use your results to calculate the enthalpy change for the reaction.



FB 1 is sodium hydrogencarbonate, NaHCO_3 .

FB 2 is sodium carbonate, Na_2CO_3 .

FB 3 is 2.0 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Step 1



- Place the plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 25 cm^3 of the acid, **FB 3**, into the plastic cup. The acid is in excess.
- Weigh the container with **FB 1** and record the balance reading in a suitable form below.
- Place the thermometer in the acid and record the initial temperature in the table of results. Tilt the cup if necessary so that the bulb of the thermometer is fully covered. This is the temperature at time zero. Start timing.
- Record the temperature of the acid at 1 minute and at 2 minutes.
- At $2\frac{1}{2}$ minutes carefully tip all the **FB 1**, in small portions to avoid spray, into the acid and stir to dissolve it.
- Record the temperature of the solution at 3, 4, 5, 6, 7 and 8 minutes.
- Reweigh the container with any residual **FB 1** and record the balance reading and the mass of **FB 1** used.
- Rinse out the plastic cup and shake it to remove excess water.

Results

Mass

I	
II	
III	
IV	
V	

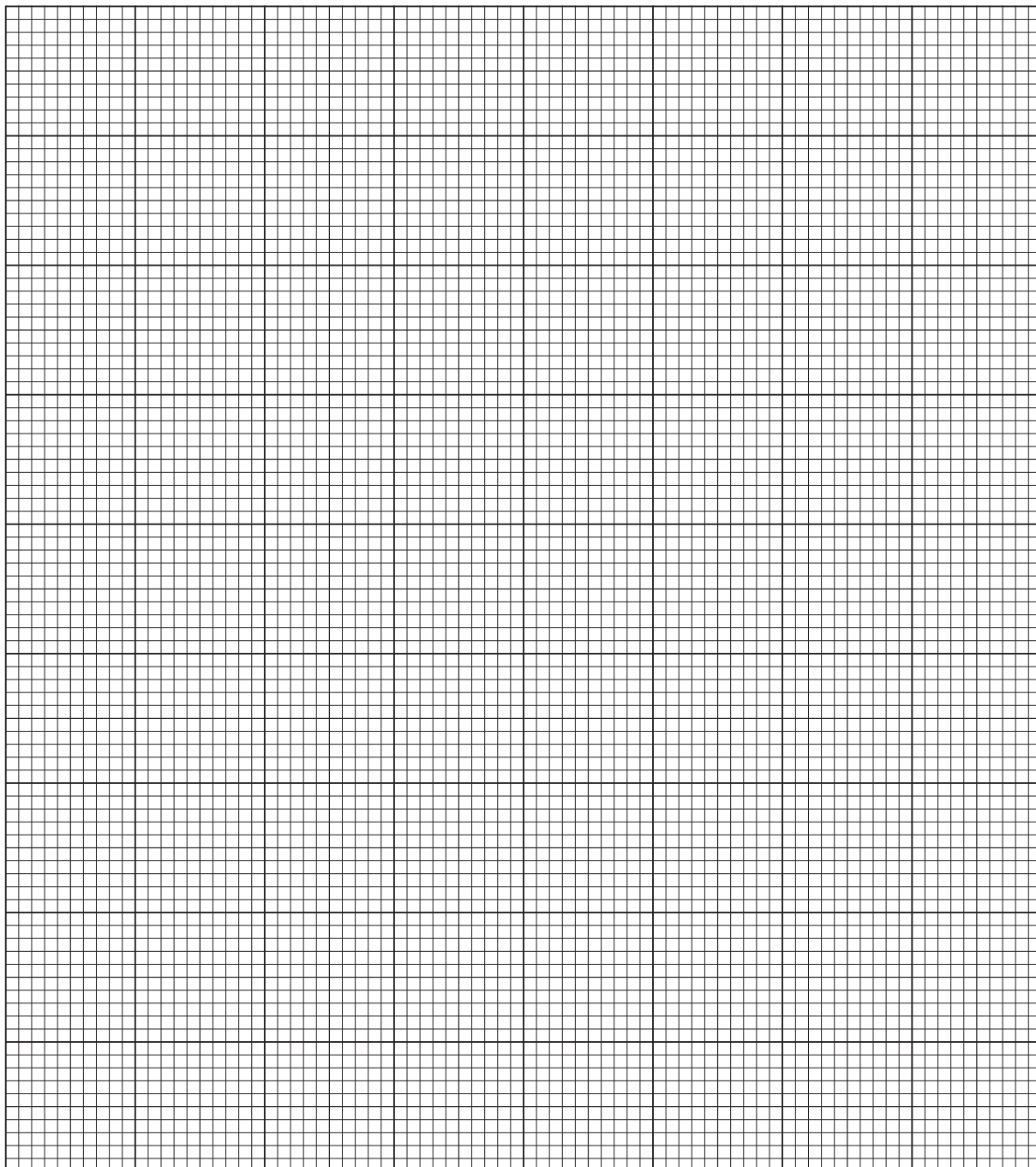
Temperature

Time in minutes	0	1	2	3	4	5	6	7	8
Temperature/ $^{\circ}\text{C}$									

[5]

- (b) Plot temperature on the y-axis against time on the x-axis on the grid below. You will use the graph to determine the theoretical temperature change at $2\frac{1}{2}$ minutes. The scale for temperature should extend at least $1\text{ }^{\circ}\text{C}$ below your lowest recorded temperature.

For
Examiner's
Use



Draw two straight lines of best fit on your graph, one for the temperature of the acid before adding **FB 1** and the other for the warming of the solution once the reaction is complete. Extrapolate the two lines to $2\frac{1}{2}$ minutes and determine the change in temperature at this time.

temperature change at $2\frac{1}{2}$ minutes = ... $^{\circ}\text{C}$
[4]

I	
II	
III	
IV	

(c) Method**Step 2**

- Read through the method and prepare a suitable table for your results.
- Place the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of the acid, **FB 3**, into the plastic cup. The acid is in excess.
- Weigh the container with **FB 2** and record the balance reading.
- Place the thermometer in the acid and record the initial temperature. Tilt the cup if necessary so that the bulb of the thermometer is fully covered.
- Carefully tip all the **FB 2**, in small portions to avoid spray, into the acid and stir to dissolve it.
- Record the highest temperature.
- Reweigh the container with any residual **FB 2** and record the balance reading and the mass of **FB 2** used.

Results

I	
II	
III	

[3]

(d) Calculations

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

- (i) Using your answer to (b), calculate the heat energy absorbed when **FB 1** was added to the acid in **step 1**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy absorbed = ... J

- (ii) Calculate the enthalpy change, in kJ mol^{-1} , when 1 mole of **FB 1**, NaHCO_3 , reacts with acid. (A_r : H, 1.0; C, 12.0; O, 16.0; Na, 23.0)

enthalpy change = ... kJ mol^{-1}
(sign) (value)

- (iii) Using your results from (c), calculate the heat energy produced when **FB 2** was added to the acid in **step 2**.

(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm^3 of solution by 1.0°C .)

heat energy produced = ... J

- (iv) Calculate the enthalpy change, in kJ mol^{-1} , when 1 mole of **FB 2**, Na_2CO_3 , reacts with acid. (A_r : C, 12.0; O, 16.0; Na, 23.0)

enthalpy change = ... kJ mol^{-1}
(sign) (value)

- (v) Use your answers to parts (ii) and (iv) and the equations for **steps 1** and **2** to determine the enthalpy change for the reaction below.



enthalpy change, ΔH = ... kJ mol^{-1}
(sign) (value) [7]

I	
II	
III	
IV	
V	
VI	
VII	

(e) (i) Complete the following table.

The balance used in the experiment reads to decimal place(s).
The maximum error in a single balance reading is g.
The maximum error in measuring the recorded mass of FB 2 in (c) would be g.

(ii) Calculate the maximum percentage error in the mass of **FB 2** in **step 2**.

maximum percentage error in the mass of **FB 2** in **step 2** = ... %
[2]

(f) (i) A student stated that the experiment could be made more accurate by using twice the volume of acid in **step 1** and **step 2**. Suggest whether the student is correct or incorrect and justify your answer.

The student is

because

.....

.....

[1]

(ii) Another student carried out **step 2** twice using the carbonate of a different metal and obtained the following results.

First result: mass used = 2.96 g; increase in temperature = 4.0 °C

Second result: mass used = 3.65 g; increase in temperature = 5.0 °C

The student then used the mean mass and the mean temperature increase when calculating the enthalpy change for the reaction. Explain whether or not the student was justified in using the results in this way, by showing appropriate calculations.

.....

.....

.....

.....

[2]

[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 name or correct formula of the element or compound must be given.

- (a) **FB 4, FB 5 and FB 6** are aqueous solutions of salts. Each contains a different anion which is either sulfate, chloride or nitrate. Using the Qualitative Analysis Notes on page 11, select reagents to allow you to identify positively which anion is in each salt using the minimum number of tests. Record your reagents and your observations in the table below.

Indicate where a test is unnecessary using a dash, —.

reagent(s)	FB 4	FB 5	FB 6

I	
II	
III	
IV	
V	

[5]

- (b) Many cations are identified by using aqueous sodium hydroxide and aqueous ammonia in small amounts and then to excess. Carry out the following tests and record all your observations in the table provided below.

<i>test</i>	<i>observations</i>		
	FB 4	FB 5	FB 6
(i) To 1 cm depth of salt solution in a test-tube, add a few drops of aqueous sodium hydroxide,			
then add excess.			
(ii) To 1 cm depth of salt solution in a test-tube, add a few drops of aqueous ammonia,			
then add excess.			

[3]

- (c) From your observations in (a) and (b), identify as many of the ions present as possible.

	FB 4	FB 5	FB 6
cation			
anion			

[3]

(d) One or more of the solutions contain(s) a second cation, the hydrogen ion, H^+ .

(i) State two tests that can be used to show whether H^+ is present.

test 1

test 2

(ii) Carry out your tests and record your observations in a suitable form in the space below.

I	
II	
III	
IV	
V	

(iii) Identify which of the solutions **FB 4**, **FB 5** and **FB 6** contain(s) the hydrogen ion, H^+ .

H^+ is present in

[5]

[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)	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), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
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NUMBER

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CHEMISTRY

9701/33

Advanced Practical Skills 1

May/June 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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.

Electronic calculators may be used.

You may lose marks if you do not show your working or if you do not use appropriate units.

Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 12 and 13.

A Periodic Table is printed on page 16.

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	

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1	
2	
3	
Total	

This document consists of **14** printed pages and **2** blank pages.



- 1 You are to determine the enthalpy change of the reaction between hydrochloric acid and sodium hydroxide by adding various volumes of acid and alkali and measuring the change in temperature.

FA 1 is $0.950 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .

FA 2 is aqueous sodium hydroxide, NaOH .

(a) Method

- Support the plastic cup in a 250 cm^3 beaker.
- Using a measuring cylinder, transfer 25 cm^3 of **FA 1** into the cup and measure the temperature of the acid. Tilt the cup if necessary to cover the bulb of the thermometer.
- Record this initial temperature.

initial temperature of **FA 1** = ... $^{\circ}\text{C}$

- Use a second measuring cylinder to transfer 10 cm^3 of **FA 2** and 25 cm^3 of water into a 100 cm^3 beaker.
- Add this mixture to the plastic cup and stir.
- Measure the maximum temperature reached and record this maximum temperature in the table below.
- Rinse out the plastic cup and shake it to remove excess water.
- Repeat the experiment, using the volumes of **FA 1**, **FA 2** and water shown in the table. Record the maximum temperature for each experiment.

volume FA 1 / cm^3	volume FA 2 / cm^3	volume water / cm^3	maximum temperature / $^{\circ}\text{C}$
25	10	25	
25	15	20	
25	20	15	
25	25	10	
25	30	5	
25	35	0	

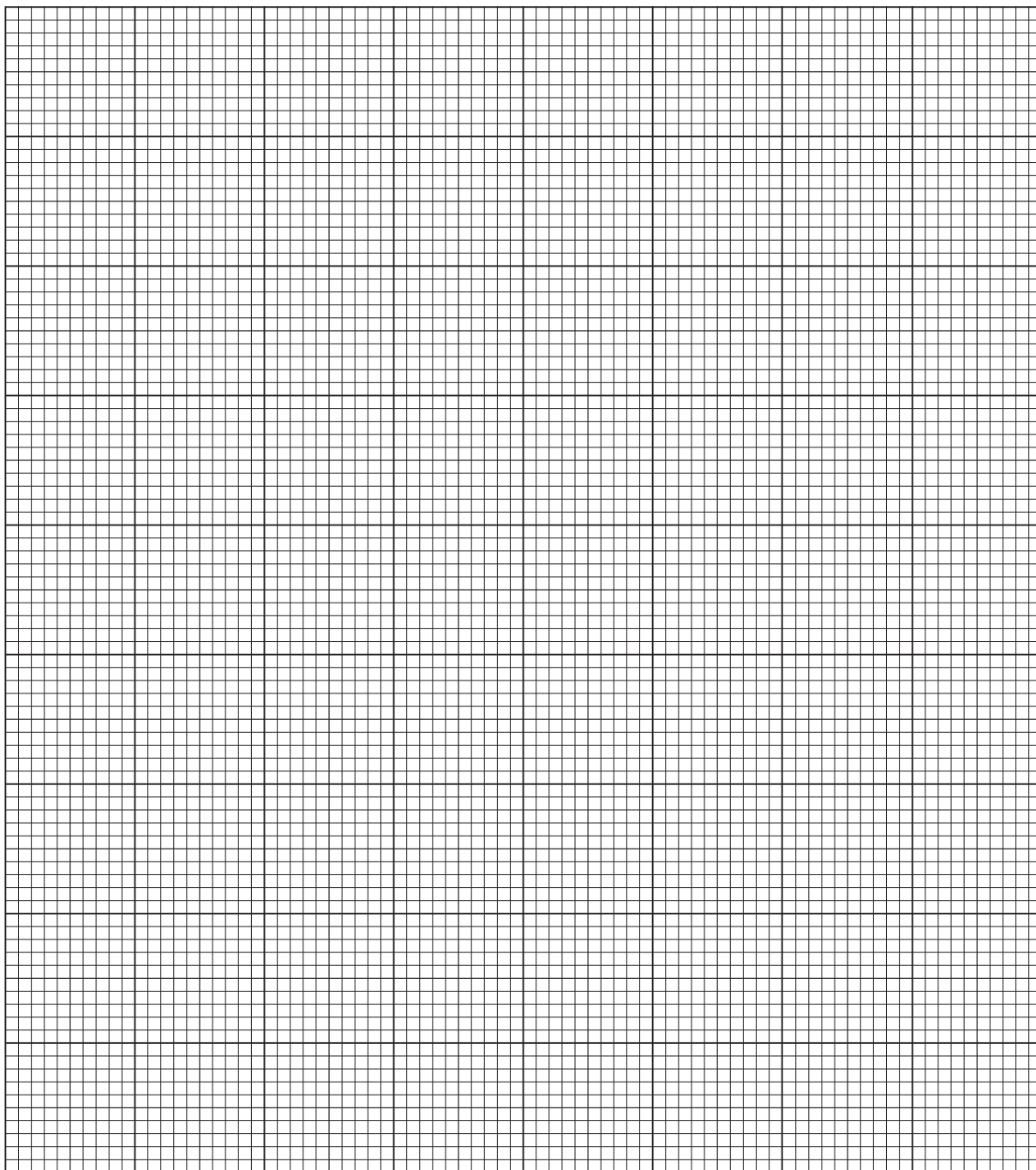
You are going to plot a graph using these results to find the volume of **FA 2** that gives the greatest maximum temperature.

Before you plot the graph, choose two further volumes of **FA 2** that will allow you to find more precisely the volume that gives the greatest maximum temperature.

Record the volumes you choose, carry out the experiments and record the corresponding maximum temperatures, in the table. [2]

- (b) (i) On the grid below, plot the maximum temperature on the y-axis against the volume of **FA 2** on the x-axis.

For
Examiner's
Use



- (ii) Draw two straight lines of best fit on your graph, one to show where the temperature was increasing and the other after the greatest maximum temperature had been reached.
- (iii) Using your graph and the initial temperature recorded in (a), determine the maximum temperature **change** that could occur when 25 cm³ of **FA 1** react with **FA 2**.

maximum temperature **change** = ... °C
[5]

(c) Calculation

- (i) Calculate the energy needed to produce the temperature change in **(b)(iii)**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

energy needed = ... J

- (ii) Calculate the number of moles of HCl used in each experiment.

moles of HCl = ... mol

- (iii) Calculate the enthalpy change, in kJ mol⁻¹, when 1 mole of HCl reacts with NaOH.

enthalpy change = ... kJ mol⁻¹
(sign) (value) [3]

[Total: 10]

- 2 The identity of a metal, M, can be found by titrating a solution of its carbonate with hydrochloric acid of known concentration.

For
Examiner's
Use

FA 3 is a solution of the metal carbonate, M_2CO_3 , of concentration 6.90 g dm^{-3} .

You are to dilute the hydrochloric acid that you used in **Question 1** and then titrate the carbonate solution with this acid.

(a) Method

Dilution of the acid

- Pipette 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric (graduated) flask labelled **FA 4**.
- Add distilled water to make the total volume 250 cm^3 .
- Stopper the flask and mix the contents thoroughly.

Titration

- Fill the burette with diluted hydrochloric acid, **FA 4**.
- Use a clean pipette to transfer 25.0 cm^3 of **FA 3** into a conical flask.
- Titrate **FA 3** with **FA 4** using the indicator provided.
- 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 4** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

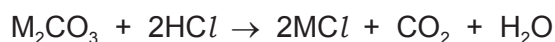
[7]

- (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 **FA 3** required cm³ of **FA 4**
[1]

(c) **Calculation**

The equation for the reaction between hydrochloric acid and the metal carbonate is given below.



- (i) Calculate the number of moles of hydrochloric acid present in the volume in (b).

moles of HCl = ... mol

- (ii) Hence, calculate the number of moles of M₂CO₃ present in 25.0 cm³ of **FA 3**.

moles of M₂CO₃ = ... mol

- (iii) Calculate the concentration of M₂CO₃ in **FA 3** in mol dm⁻³.

concentration of M₂CO₃ = ... mol dm⁻³

- (iv) Use your answer to (iii), and the fact that **FA 3** contains 6.90 g dm⁻³, to determine the relative atomic mass, *A_r*, of M.

A_r of M = ...

- (v) Use your answer to (iv) and the Periodic Table on page 16 to suggest the identity of M.

M is [5]

(d) The concentration of a carbonate solution could be found using either the method in **Question 1** or that in **Question 2**.

(i) Suggest, and explain, which of the methods is more accurate.

.....

.....

(ii) For the method that you think is less accurate, suggest an improvement to the practical procedure that could be made to improve the accuracy.

.....

..... [2]

[Total: 15]

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.

- (a) You are provided with a solid, **FA 5**. **FA 5** is a mixture that contains two anions and two cations.

To all your sample of **FA 5** in a boiling tube add 3 cm depth of distilled water. Shake the tube and filter the contents. Keep the solid residue for tests in (b) and the filtered solution for tests in (c).

- (b) (i) Open up the filter paper and scrape the residue into a boiling tube. Add dilute nitric acid, HNO_3 , using a dropping pipette until the solid **just** disappears. Record your observations and keep the solution for tests in (ii).

observations

.....

- (ii) Divide the solution from test (i) equally into **three** test-tubes.

To the first test-tube add aqueous sodium hydroxide, NaOH , until in excess. Record your observations.

observations

.....

Which cations, from those listed in the Qualitative Analysis Notes on page 12, would give these observations?

.....

- (iii) You are to devise tests that will positively identify which one of the cations you have suggested in (ii) is present. For each of the possible ions you should indicate the test and the expected result for each test in a suitable table in the space below.

Use the solutions in the second and third test-tubes to carry out these tests and record your observations in the space below.

Identify the cation present.

The cation present is

[7]

- (c) To 1 cm depth of filtered solution from (a) in a test-tube add 1 cm depth of dilute nitric acid followed by a few drops of aqueous silver nitrate. Record your observation.

observation

Which further reagent could be added to this test-tube to help you to confirm the nature of the anion present?

reagent

Carry out a test using this additional reagent. Record your observation and conclusion about the anion present.

observation

The anion present is [2]

- (d) Using your observation in (b)(i) state which other anion is present in **FA 5**.

The anion present is [1]

- (e) Solutions **FA 6** and **FA 7** each contain one of the ions sulfite, SO_3^{2-} , sulfate, SO_4^{2-} , nitrite, NO_2^- , or nitrate, NO_3^- .

- (i) Carry out the tests in the table below to identify which ion is present in each solution.

test	observations	
	FA 6	FA 7
To 1 cm depth of solution in a boiling tube, add a small piece of aluminium foil and 1 cm depth of aqueous sodium hydroxide. Warm the mixture with care .		
To 1 cm depth of solution in a test-tube, add a few drops of aqueous barium chloride or barium nitrate, then		
add dilute hydrochloric acid.		
To 1 cm depth of solution in a test-tube, add 1 cm depth of dilute hydrochloric acid.		

- (ii) From your observations, identify the anion present in each solution.

FA 6 contains

FA 7 contains

- (iii) What type of reaction takes place when a positive observation is seen with aluminium foil and aqueous sodium hydroxide in (i)?

.....
[5]

[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)	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), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

[illegible]

9701/33/M/J/13



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CHEMISTRY

9701/34

Advanced Practical Skills 2

May/June 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 12 and 13.

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.

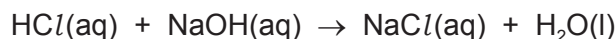
Session	
Laboratory	

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1	
2	
3	
Total	

This document consists of **12** printed pages and **4** blank pages.

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- 1 When aqueous hydrochloric acid is mixed with aqueous sodium hydroxide, the neutralisation reaction releases heat causing a rise in the temperature of the solution.



In this experiment you will mix different volumes of hydrochloric acid and sodium hydroxide but the total volume will be kept constant. For each mixture you will record the temperature rise. Since the combined volume remains the same, the temperature rise is a direct measure of the heat given out by the reaction. The maximum heat given out occurs when all the acid present is exactly neutralised by all the alkali present. By determining the volumes when this occurs you can work out the concentration of the sodium hydroxide.

FB 1 is 2.00 mol dm⁻³ hydrochloric acid, HCl.

FB 2 is aqueous sodium hydroxide, NaOH.

Read through the instructions carefully and prepare a table for your results in the space on page 4 before starting any practical work.

(a) Method

Experiment 1

- Support the plastic cup in the 250 cm³ beaker.
- Fill the unlabelled burette with **FB 1**.
- Run 26.00 cm³ of **FB 1** from the burette into the plastic cup.
- Record the temperature of **FB 1**, T_1 , in the space below.

$T_1 = \dots\dots\dots$ °C

- Fill the burette labelled **FB 2** with **FB 2**.
- Run 4.00 cm³ of **FB 2** from the burette into the plastic cup.
- Stir the mixture thoroughly and record in your table the maximum temperature of the solution.
- Empty the plastic cup, rinse thoroughly with water and shake dry.

Experiment 2

- Support the plastic cup in the 250 cm³ beaker.
- Run 22.00 cm³ of **FB 1** from the burette into the plastic cup.
- Run 8.00 cm³ of **FB 2** from the burette into the plastic cup.
- Stir the mixture thoroughly and record in your table the maximum temperature of the solution.
- Empty the plastic cup, rinse thoroughly with water and shake dry.

Experiments 3 – 7

- Repeat the experiment using 18.00, 14.00, 10.00, 6.00 and 2.00 cm³ of **FB 1** respectively. Add sufficient **FB 2** each time to make sure that the total volume remains 30.00 cm³.

For **each** of your **seven** experiments, record in the space below

- the volume of **FB 1**,
- the volume of **FB 2**,
- the maximum temperature of the solution,
- the temperature rise, ΔT , where $\Delta T = \text{maximum temperature recorded} - T_1$.

I	
II	
III	
IV	
V	
VI	

[6]

- (b) (i) On the grid opposite, plot the temperature rise, ΔT , on the y-axis against the volume of **FB 1** on the x-axis.
The scale for ΔT should extend at least 2 °C above your greatest temperature rise.

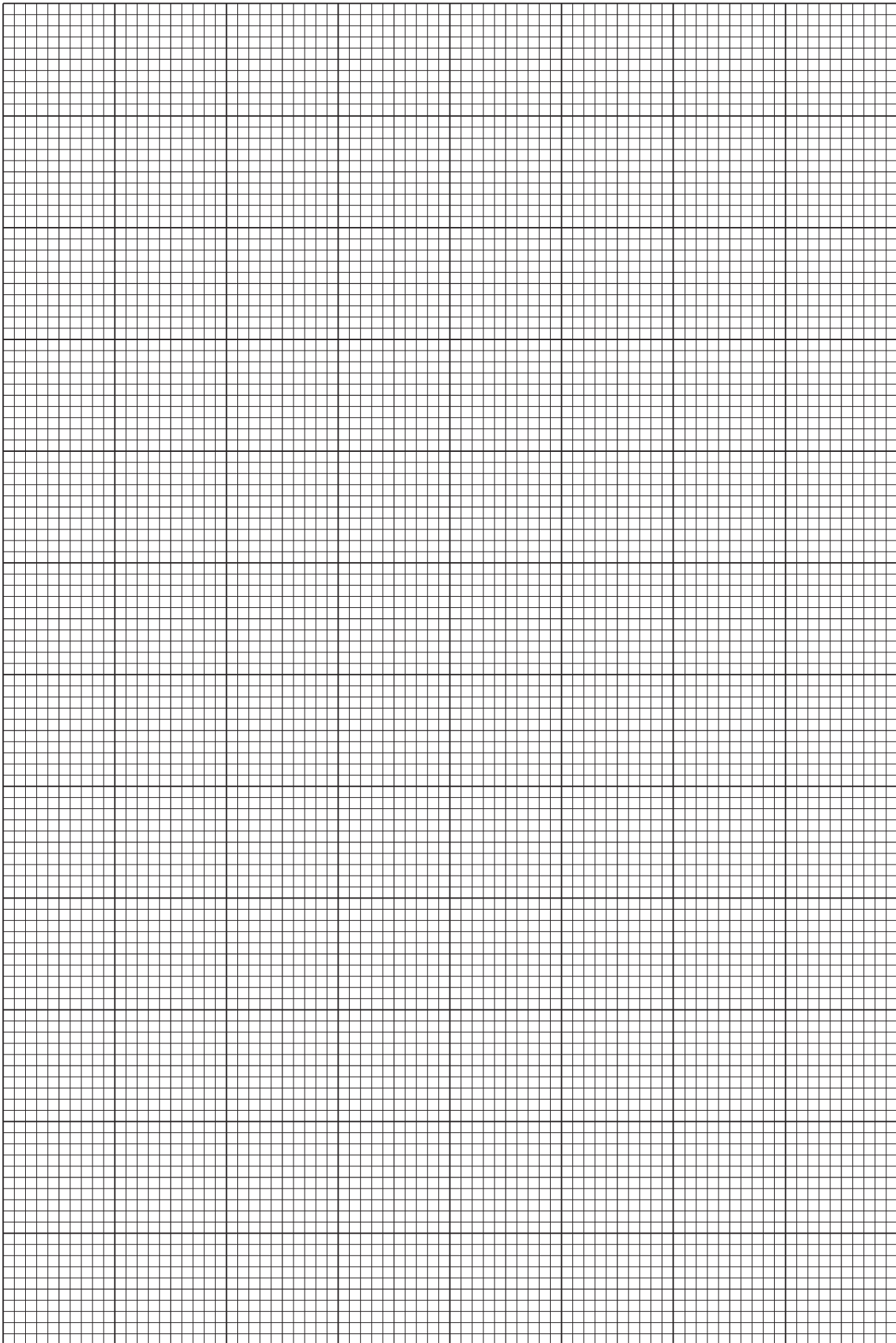
- (ii) Draw a straight line of best fit through the points where the values of ΔT are increasing.
Draw a second straight line of best fit through the points where the values of ΔT are decreasing.

- (iii) From your graph, determine the value of the volume of **FB 1** where the two lines of best fit intersect.

I	
II	
III	
IV	
V	

volume of **FB 1** = ... cm³

[5]



(c) Calculations

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

- (i) Calculate how many moles of hydrochloric acid are contained in the volume recorded in **(b)(iii)**.

moles of HCl = ... mol

- (ii) Calculate how many moles of sodium hydroxide would react completely with the number of moles of hydrochloric acid in **(c)(i)**.

moles of NaOH = ... mol

- (iii) Calculate the concentration of **FB 2**. Remember that the combined volume of **FB 1** and **FB 2** in each experiment was 30.00 cm^3 .

concentration of **FB 2** = ... mol dm^{-3}
[3]

- (d) A student decided to modify the experiment. The total volume of the solution was increased to 50 cm^3 and temperature rises were recorded for 5, 10, 15, 20, 25, 30, 35, 40 and 45 cm^3 of **FB 2**. The volumes were measured using a 50 cm^3 measuring cylinder. Discuss how these changes would affect the accuracy with which the concentration of **FB 2** could be determined.

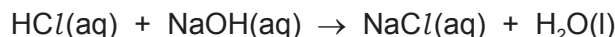
.....
.....
..... [2]

[Total: 16]

I	
II	
III	

- 2 A second way to determine the concentration of an alkali is by volumetric titration. In this experiment you will first dilute the sample of **FB 2** that you used in **Question 1** and then titrate the diluted solution using hydrochloric acid.

For
Examiner's
Use



FB 2 is aqueous sodium hydroxide, NaOH.

FB 3 is $0.200 \text{ mol dm}^{-3}$ hydrochloric acid, HCl.

(a) Method

Dilution of FB 2

- Use the burette labelled **FB 2** to transfer 25.00 cm^3 of **FB 2** into the 250 cm^3 graduated (volumetric) flask, labelled **FB 4**.
- Make up the contents of the flask to the 250 cm^3 mark with distilled water.
- Stopper the flask and mix the contents thoroughly. This solution **FB 4**.

Titration

- Rinse the unlabelled burette thoroughly with distilled water and then with a little **FB 3**. Fill this burette with **FB 3**.
- Use a pipette to transfer 25.0 cm^3 of **FB 4** into a conical flask.
- Add to the flask a few drops of the acid-base indicator provided.
- Titrate the alkali in the flask with the acid, **FB 3**.

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 **FB 3** added in each accurate titration. Make certain that any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	

[5]

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

For
Examiner's
Use

25.0 cm³ of **FB 4** required cm³ of **FB 3**.
[1]

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

moles of HCl = ... mol

- (ii) Hence, calculate how many moles of NaOH are contained in 25.0 cm³ of **FB 4**.

moles of NaOH = ... mol

- (iii) Calculate the concentration of the sodium hydroxide, **FB 2**.

concentration of **FB 2** = ... mol dm⁻³
[3]

[Total: 9]

I	
II	
III	

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.

(a) **FB 5, FB 6, FB 7 and FB 8** are aqueous solutions each of which contains a single cation and a single anion. Some of the ions present are listed below.



By observing the reactions that occur when pairs of the solutions are mixed together, you will be able to identify which solution contains which of these ions.

- To a 1 cm depth of **FB 6**, **FB 7** and **FB 8** in separate test-tubes add **FB 5** dropwise until in excess. Leave each test-tube to stand for a few minutes and note any changes.
- Use a 1 cm depth of each solution for the remaining tests.
- Record your observations in the following table.

For
Examiner's
Use

	FB 6	FB 7	FB 8
FB 5			
FB 6			
FB 7			

[6]

I	
II	
III	
IV	
V	
VI	

(b) From your observations deduce which solution contains each of the following ions.

ion	Fe ²⁺	Pb ²⁺	Zn ²⁺	I ⁻	OH ⁻	SO ₄ ²⁻
solution						

[3]

I	
II	
III	

- (c) **FB 9** is an aqueous solution containing either sulfate, SO_4^{2-} , or sulfite, SO_3^{2-} , ions. Describe how you would determine which ion is present.

.....

Carry out this test and record your observations and conclusion.

observations

The anion in **FB 9** is [2]

- (d) A student is given two **different** samples and told that each contains one of the following organic compounds.

ethanol

ethanal

ethanoic acid

Describe two chemical tests that the student could carry out on each sample to determine which organic compound is present in each sample. For each test, give the reagent(s) to be used and explain how the different observations would allow the identity of the sample to be determined.

first reagent

observations

explanation

second reagent

observations

explanation

[4]

[Total: 15]

I	
II	
III	
IV	

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)	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), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

CANDIDATE
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NUMBER

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CANDIDATE
NUMBER

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CHEMISTRY

9701/35

Advanced Practical Skills 1

May/June 2013

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 10 and 11.

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.

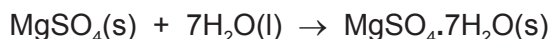
Session
Laboratory

For Examiner's Use	
1	
2	
Total	

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



- 1 You will determine the enthalpy change, ΔH , for the reaction of anhydrous magnesium sulfate, MgSO_4 , with water to form hydrated magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. In step 1 you will dissolve a known mass of anhydrous magnesium sulfate in a known volume of water and find the temperature change. In step 2 you will find the temperature change on adding a known mass of hydrated magnesium sulfate to a known volume of water. You will then use your results to calculate the enthalpy change for the reaction.



FA 1 is anhydrous magnesium sulfate, MgSO_4 .

FA 2 is hydrated magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

distilled water

(a) Method

Step 1



- Place the plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 25 cm^3 of distilled water into the plastic cup.
- Weigh the container with **FA 1** and record the balance reading in a suitable form in the space below.
- Place the thermometer in the water and record the initial temperature in the table of results. Tilt the cup if necessary so that the bulb of the thermometer is fully covered. This is the temperature at time zero. Start timing.
- Record the temperature of the water at 1 minute and at 2 minutes.
- At $2\frac{1}{2}$ minutes tip all the **FA 1** into the water and stir to dissolve.
- Record the temperature of the solution at 3, 4, 5, 6, 7 and 8 minutes.
- Reweigh the container with any residual **FA 1** and record the balance reading and the mass of **FA 1** used.
- Rinse out the plastic cup and shake it to remove excess water.

Results

Mass

Temperature

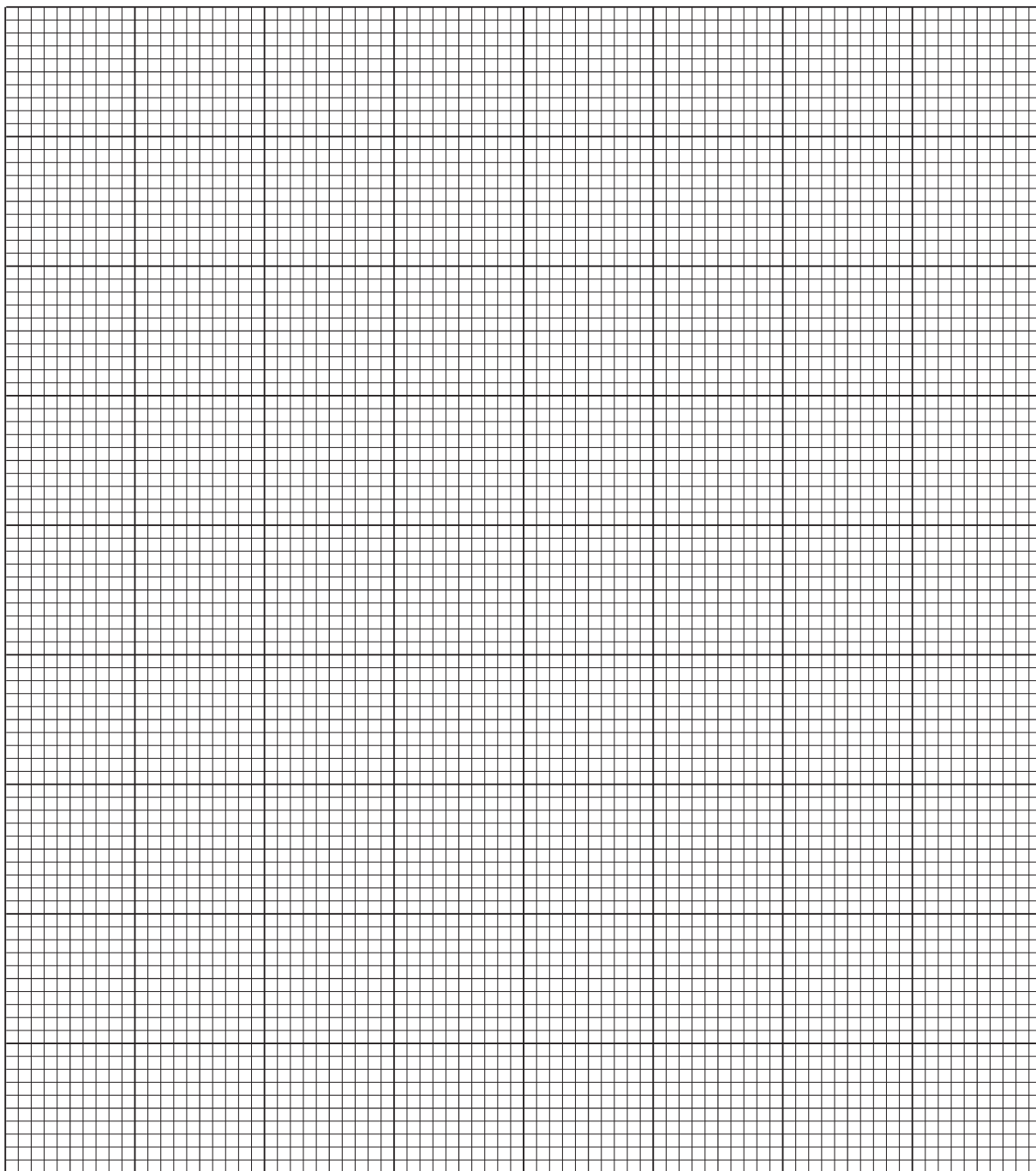
Time in minutes	0	1	2	3	4	5	6	7	8
Temperature / °C									

[5]

I	
II	
III	
IV	
V	

- (b) Plot temperature on the y-axis against time on the x-axis on the grid below. You will use the graph to determine the theoretical temperature change at $2\frac{1}{2}$ minutes.

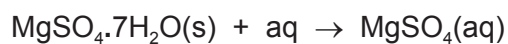
For
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Use



Draw two straight lines of best fit on your graph, one for the temperature of the water before adding **FA 1** and the other for the cooling of the solution once the reaction is complete. Extrapolate the two lines to $2\frac{1}{2}$ minutes and determine the change in temperature at this time.

temperature change at $2\frac{1}{2}$ minutes = ... °C
[4]

I	
II	
III	
IV	

(c) Method**Step 2**

- Read through the method and prepare a suitable table for your results.
- Place the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of distilled water into the plastic cup.
- Weigh the container with **FA 2** and record the balance reading below.
- Place the thermometer in the water and record the initial temperature. Tilt the cup if necessary so that the bulb of the thermometer is fully covered.
- Tip all the **FA 2** into the water and stir to dissolve.
- Record the lowest temperature.
- Reweigh the container with any residual **FA 2** and record the balance reading and the mass of **FA 2** used.

Results

I	
II	
III	

[3]

(d) Calculations

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

- (i) Using your answer to (b), calculate the heat energy produced when **FA 1** was added to water in **step 1**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy produced = ... J

- (ii) Calculate the enthalpy change, in kJ mol⁻¹, when 1 mole of **FA 1**, MgSO₄, is dissolved.
(A_r: O, 16.0; Mg, 24.3; S, 32.1)

enthalpy change = ... kJ mol⁻¹
(sign) (value)

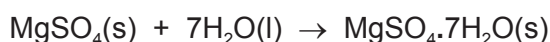
- (iii) Using your results from (c), calculate the heat energy absorbed when **FA 2** was added to water in **step 2**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

heat energy absorbed = ... J

- (iv) Calculate the enthalpy change, in kJ mol⁻¹, when 1 mole of **FA 2**, MgSO₄·7H₂O, is dissolved.
(A_r: H, 1.0; O, 16.0; Mg, 24.3; S, 32.1)

enthalpy change = ... kJ mol⁻¹
(sign) (value)

- (v) Use your answers to parts (ii) and (iv) and the equations for the reactions shown in **steps 1** and **2** to determine the enthalpy change, in kJ mol⁻¹, for the reaction below.



enthalpy change, ΔH = ... kJ mol⁻¹
(sign) (value) [6]

I	
II	
III	
IV	
V	
VI	

(e) (i) Complete the following table.

The maximum error in a single thermometer reading is °C.
The maximum error in measuring the change in temperature in step 2 is °C.

(ii) Calculate the maximum percentage error in the temperature change in **step 2**.

maximum percentage error in the temperature change in **step 2** = ... %
[2]

(f) (i) A student suggested that the experiment could be made more accurate by using 50 cm³ of water in **step 1** and **step 2**. State whether the student is correct or incorrect and justify your answer.

The student is

because

[1]

(ii) Another student carried out **step 2** twice for a different hydrated salt and obtained the following results.

First result: mass used = 3.34 g; drop in temperature = 4.0 °C

Second result: mass used = 4.18 g; drop in temperature = 5.0 °C

The student then used the mean mass and mean temperature drop when calculating the enthalpy change for the reaction. Explain whether or not the student was justified in using the results in this way, by showing appropriate calculations.

[2]

[Total: 23]

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 name or correct formula of the element or compound must be given.

FA 3, FA 4 and FA 5 are aqueous solutions of salts. Read through the tests before starting the practical work.

- (a)** Many cations are identified by using aqueous sodium hydroxide and aqueous ammonia in small amounts and then to excess. Carry out the following tests and record all your observations in the table below.

Do **not** discard the final products in these tests as you will need to use them in part **(b)**.

<i>test</i>	<i>observations</i>		
	FA 3	FA 4	FA 5
(i) To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide,			
until in excess.			
(do not discard)			
(ii) To 1 cm depth of solution in a test-tube, add aqueous ammonia,			
until in excess.			
(do not discard)			

[3]

(b) At least one of the solutions contains a second cation, the ammonium ion, NH_4^+ . Devise a test to identify which salt or salts contain the ammonium ion. You are to use the products of the reactions in **(a)** when carrying out your test.

(i) Which of the following sets of products will you use in your test? Tick the appropriate box.

- products with excess aqueous sodium hydroxide ☐
- products with excess aqueous ammonia ☐

(ii) Describe the test and expected observations if NH_4^+ is present.

.....

.....

.....

(iii) Carry out your test and record your observations clearly in the space below.

[4]

- (c) **FA 3**, **FA 4** and **FA 5** each contains a different anion which is sulfate, chloride, or nitrate. Using the Qualitative Analysis Notes on page 11, select reagents to allow you to identify positively which anion is in each salt using the minimum number of tests. Record your reagents and your observations in the table below.

Indicate where a test is unnecessary using a dash, —.

reagent(s)	FA 3	FA 4	FA 5

[5]

- (d) From your observations in (a), (b) and (c), identify as many ions that could be present as possible.

	FA 3	FA 4	FA 5
cation(s)			
anion			

[5]

[Total: 17]

For
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Use

I	
II	
III	
IV	
V	

I	
II	
III	
IV	
V	

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)	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), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

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