



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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CANDIDATE
NUMBER

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CHEMISTRY

9701/31

Advanced Practical Skills 1

October/November 2012

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.

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

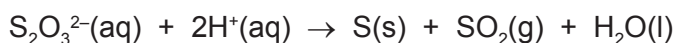
For Examiner's Use	
1	
2	
Total	

This document consists of **13** printed pages and **3** blank pages and **1** insert.



- 1 You are to investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid changes as the concentration of the sodium thiosulfate solution is varied.

When aqueous sodium thiosulfate reacts with aqueous hydrogen ions present in an acid, a fine suspension of solid sulfur is formed in the solution.



The rate of reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur. The beaker containing a constant volume of reaction mixture is placed on the printed insert supplied. The time is recorded when the print is no longer visible through the suspension of sulfur.

FA 1 is 0.150 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃.

FA 2 is 0.500 mol dm⁻³ hydrochloric acid, HCl.
distilled water

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

In each of the following experiments you will use varying volumes of **FA 1** and distilled water and a fixed volume of **FA 2**. The **total** volume of solution used will be constant.

(a) Method

Experiment 1

- Use the measuring cylinder labelled **A** to transfer 50 cm³ of **FA 1** into a 250 cm³ beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm³ of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
- View the print from directly above and through the solution in the beaker.
- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

Experiment 2

- Use the measuring cylinder labelled **A** to transfer 40 cm³ of **FA 1** into the rinsed and dried beaker.
- Use the same measuring cylinder labelled **A** to transfer 10 cm³ of distilled water into the same beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm³ of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
- View the print from directly above and through the solution in the beaker.
- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

Experiments 3–5

- Repeat the experiment using the following volumes of **FA 1** and distilled water.

Experiment 3 30 cm³ of **FA 1** + 20 cm³ of distilled water

Experiment 4 20 cm³ of **FA 1** + 30 cm³ of distilled water

Experiment 5 10 cm³ of **FA 1** + 40 cm³ of distilled water

Experiment 6

When you have completed experiments 1–5, carry out one further experiment using a different volume of both **FA 1** and distilled water.

When you have completed all of your experiments, empty and rinse the beaker.

Record the results for all six experiments in the space below.
Your table should include columns for the following.

- experiment number
- volume of **FA 1**
- volume of distilled water
- reaction time
- $\frac{(1000)}{(\text{reaction time})}$

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	
X	
XI	

[11]

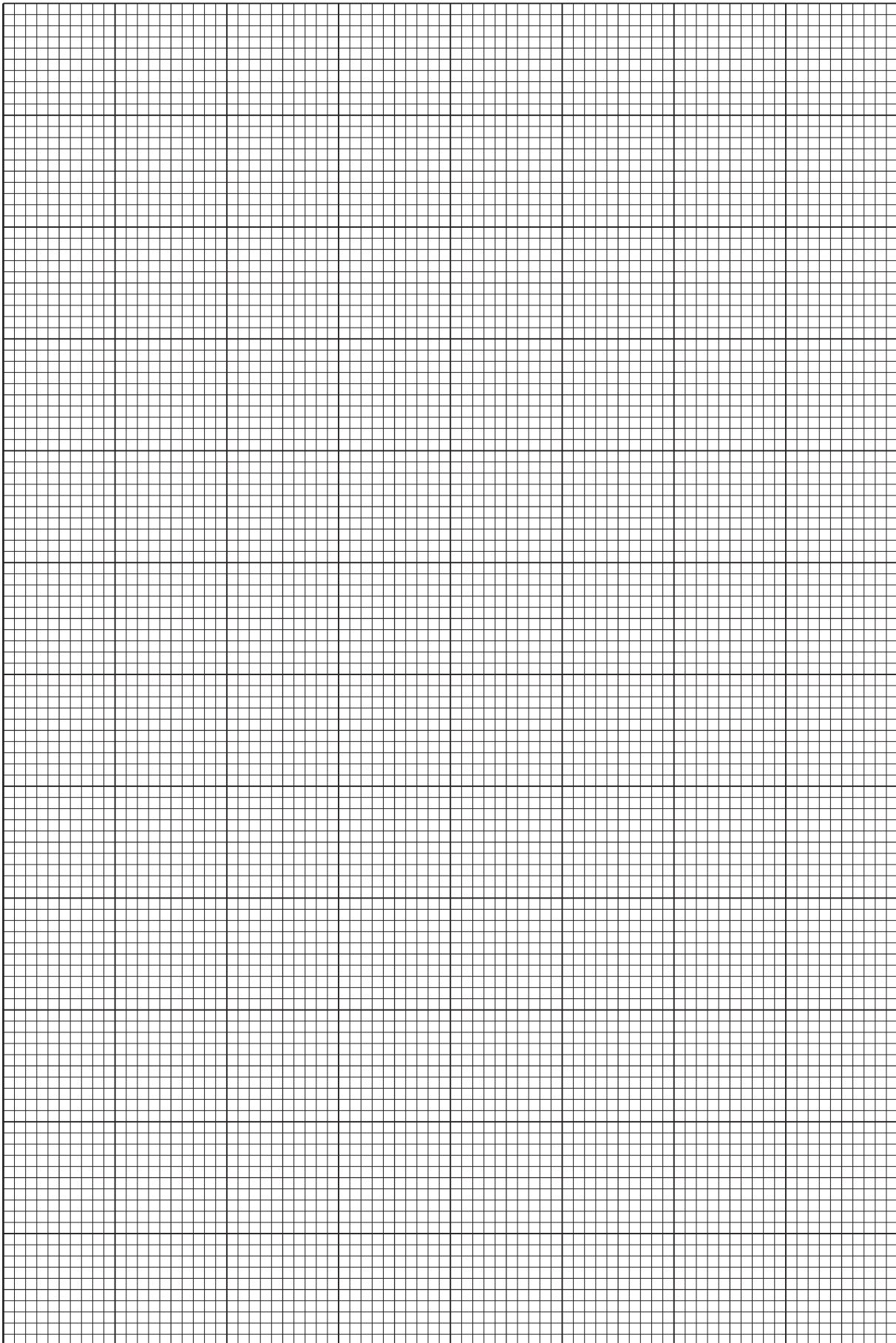
(b) The rate of reaction can be represented by the following formula.

$$\text{'rate'} = \frac{(1000)}{(\text{reaction time})}$$

On the next page plot a graph of '**rate**' against the **volume of FA 1**.

Start each of the axes at zero.

Draw the line of best fit.



I	
II	
III	
IV	
V	

[5]

- (c) A student carried out the experiments in a 100 cm³ beaker instead of a 250 cm³ beaker. State and explain what effect this would have on the times recorded.

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

- (d) **FA 1** is 0.150 mol dm⁻³ Na₂S₂O₃.
Calculate the initial concentration of Na₂S₂O₃ in the reaction mixture in **Experiment 5**.
Show your working.

The initial concentration of Na₂S₂O₃ in **Experiment 5** = ... mol dm⁻³
[2]

- (e) In your experiments, the volume of **FA 1** represents the initial concentration of sodium thiosulfate in the reaction mixture.
A text book states that the rate of reaction between aqueous sodium thiosulfate and hydrochloric acid is directly proportional to the concentration of sodium thiosulfate.
Use your graph to decide whether the statement in the text book is correct or not.
Explain your answer.

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

- (f) When viewing the insert through the solution it is difficult to judge the exact moment when the printed material just disappears.

This uncertainty is different for each experiment and is greater for longer reaction times when the printed material disappears slowly.

Complete the table below, assuming the uncertainties given.

	Experiment 1	Experiment 5
recorded reaction time/s		
uncertainty/s	± 2	± 8
percentage uncertainty	%	%

[1]

- (g) Complete the headings in the table below to record the volume of **FA 1** (aqueous sodium thiosulfate), the volume of distilled water and the volume of **FA 2** (hydrochloric acid).

In the second row copy the volumes used in **Experiment 3** from your table of results on page 4.

In the following two rows suggest volumes of each of the reagents that could be used in two further experiments, **Experiment 7** and **Experiment 8**, to investigate how the rate of reaction varies with a change in the concentration of the acid.

Do not carry out these experiments.

Experiment			
3			
7			
8			

[2]

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

Solutions **FA 3**, **FA 4**, **FA 5** and **FA 6** each contain one cation and one anion from those listed on pages 12 and 13.

- (a) Some cations interfere with tests for anions and have to be removed from the solution before the tests for anions present can be performed. One way in which this can be carried out is to precipitate the cation in the form of its insoluble carbonate.

Carry out the following tests on both **FA 3** and **FA 5**.

<i>test</i>	<i>observation</i>	
	FA 3	FA 5
To 5 cm depth of solution in a boiling tube, add all of the sodium carbonate, Na_2CO_3 , from one of the tubes provided.		
Stir the mixture.		

Retain the mixture from **FA 3** for use in (b).

[4]

- (b) Filter the mixture from **FA 3** from (a) into another boiling tube. Ignore any colour in the filtered solution.

Add 5 cm depth of dilute nitric acid. This removes any excess of carbonate ions.

Carry out the following tests on the acidified filtrate from **FA 3**.

<i>test</i>	<i>observation</i>
To 2 cm depth of the acidified filtrate from FA 3 in a test-tube, add 1 cm depth of aqueous silver nitrate, then	
add an excess of aqueous ammonia.	

[1]

- (c) Carry out the following test on **FA 4**.

<i>test</i>	<i>observation</i>
To 1 cm depth of FA 4 in a test-tube, add 1 cm depth of FA 3 , then	
add a few drops of starch solution.	

[2]

(d) Carry out the following tests.

test	observation			
	FA 3	FA 4	FA 5	FA 6
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous sodium hydroxide, then				
add a further 2 cm depth of aqueous sodium hydroxide.				
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous ammonia, then				
add a further 2 cm depth of aqueous ammonia.				

[4]

(e) The results from tests in (d) should enable you to identify either a single cation in a solution, or a pair of cations which have identical reactions with the reagents used. Identify any single ion that is present or suggest a pair of ions that may be present in each of the solutions.

solution	cation(s)
FA 3	
FA 4	
FA 5	
FA 6	

[2]

- (f) Where you were unable to identify a single cation in (e), suggest a suitable reagent which would allow you to identify which cation is present in the solution.

Do not carry out this test.

The reagent to be used is

State the expected observations to identify the presence of each of the cations from the pair of ions you have given in (e).

cation 1

.....

cation 2

..... [1]

- (g) By considering the results of **all** your tests, enter one of the following responses in each of the boxes below.

- chloride
- bromide
- iodide
- no halide ion is present
- insufficient tests (have been performed to identify any halide ion)

FA 3	
FA 4	
FA 5	

[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)	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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CHEMISTRY

9701/33

Advanced Practical Skills 1

October/November 2012

2 hours

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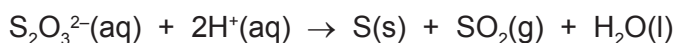
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2	
Total	

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- 1 You are to investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid changes as the concentration of the sodium thiosulfate solution is varied.

When aqueous sodium thiosulfate reacts with aqueous hydrogen ions present in an acid, a fine suspension of solid sulfur is formed in the solution.



The rate of reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur. The beaker containing a constant volume of reaction mixture is placed on the printed insert supplied. The time is recorded when the print is no longer visible through the suspension of sulfur.

FA 1 is 0.150 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃.

FA 2 is 0.500 mol dm⁻³ hydrochloric acid, HCl.
distilled water

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

In each of the following experiments you will use varying volumes of **FA 1** and distilled water and a fixed volume of **FA 2**. The **total** volume of solution used will be constant.

(a) Method

Experiment 1

- Use the measuring cylinder labelled **A** to transfer 50 cm³ of **FA 1** into a 250 cm³ beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm³ of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
- View the print from directly above and through the solution in the beaker.
- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

Experiment 2

- Use the measuring cylinder labelled **A** to transfer 40 cm³ of **FA 1** into the rinsed and dried beaker.
- Use the same measuring cylinder labelled **A** to transfer 10 cm³ of distilled water into the same beaker.
- Use the measuring cylinder labelled **B** to measure 20 cm³ of **FA 2**.
- Tip the **FA 2** from measuring cylinder **B** into the beaker and **immediately** start timing.
- Stir the contents of the beaker once and place the beaker on the printed insert.
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- Stop timing when the printed material on the insert is **just** no longer visible.
- Record the reaction time to the nearest second.
- Calculate and record the value of $\frac{(1000)}{(\text{reaction time})}$ to 3 significant figures.
- Empty, rinse and dry the beaker.

Experiments 3–5

- Repeat the experiment using the following volumes of **FA 1** and distilled water.

Experiment 3 30 cm³ of **FA 1** + 20 cm³ of distilled water

Experiment 4 20 cm³ of **FA 1** + 30 cm³ of distilled water

Experiment 5 10 cm³ of **FA 1** + 40 cm³ of distilled water

Experiment 6

When you have completed experiments 1–5, carry out one further experiment using a different volume of both **FA 1** and distilled water.

When you have completed all of your experiments, empty and rinse the beaker.

Record the results for all six experiments in the space below.
Your table should include columns for the following.

- experiment number
- volume of **FA 1**
- volume of distilled water
- reaction time
- $\frac{(1000)}{(\text{reaction time})}$

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	
X	
XI	

[11]

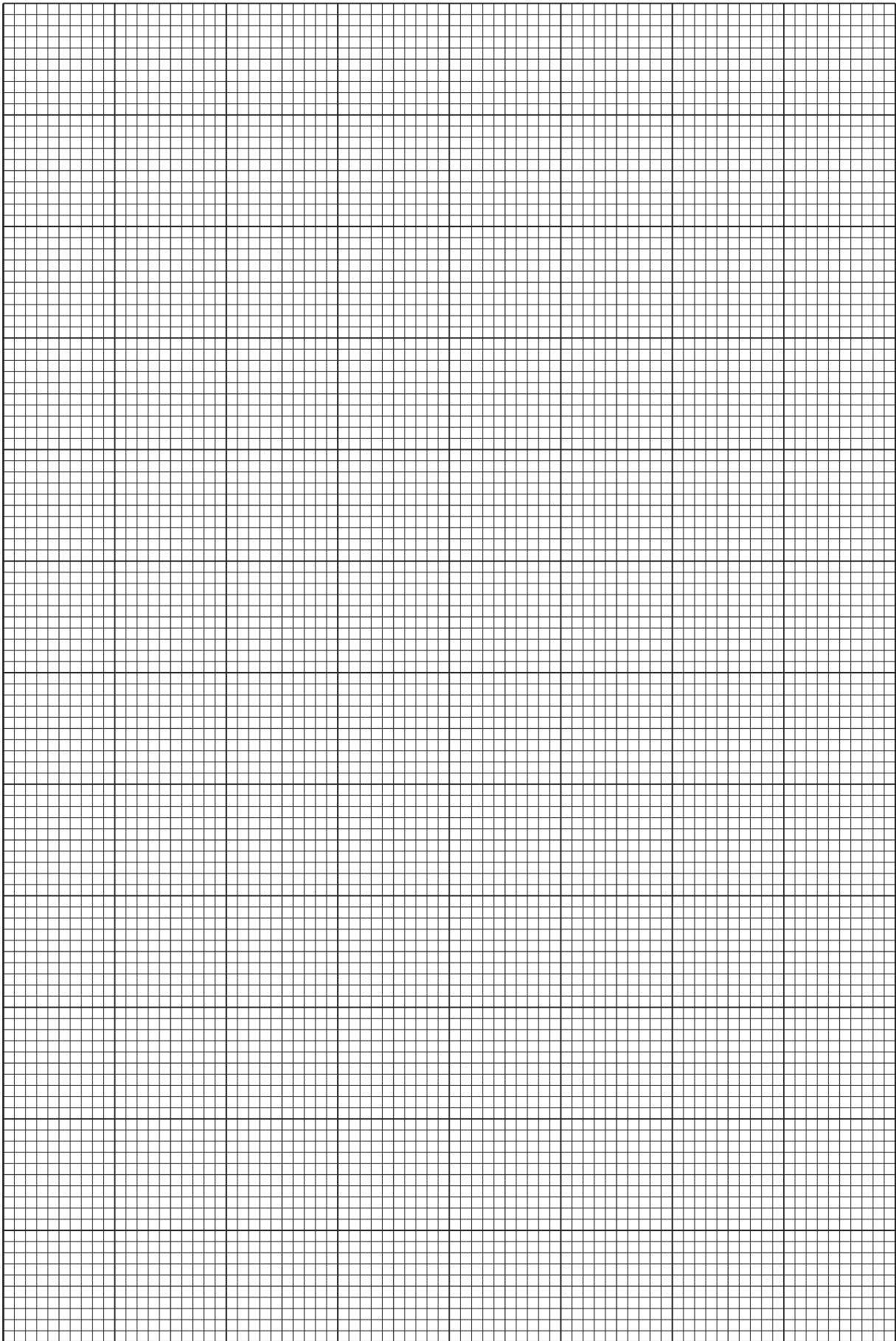
(b) The rate of reaction can be represented by the following formula.

$$\text{'rate'} = \frac{(1000)}{(\text{reaction time})}$$

On the next page plot a graph of **'rate'** against the **volume of FA 1**.

Start each of the axes at zero.

Draw the line of best fit.



I	
II	
III	
IV	
V	

[5]

- (c) A student carried out the experiments in a 100 cm³ beaker instead of a 250 cm³ beaker. State and explain what effect this would have on the times recorded.

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

- (d) **FA 1** is 0.150 mol dm⁻³ Na₂S₂O₃.
Calculate the initial concentration of Na₂S₂O₃ in the reaction mixture in **Experiment 5**.
Show your working.

The initial concentration of Na₂S₂O₃ in **Experiment 5** = ... mol dm⁻³
[2]

- (e) In your experiments, the volume of **FA 1** represents the initial concentration of sodium thiosulfate in the reaction mixture.
A text book states that the rate of reaction between aqueous sodium thiosulfate and hydrochloric acid is directly proportional to the concentration of sodium thiosulfate.
Use your graph to decide whether the statement in the text book is correct or not.
Explain your answer.

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

- (f) When viewing the insert through the solution it is difficult to judge the exact moment when the printed material just disappears.

This uncertainty is different for each experiment and is greater for longer reaction times when the printed material disappears slowly.

Complete the table below, assuming the uncertainties given.

	Experiment 1	Experiment 5
recorded reaction time/s		
uncertainty/s	± 2	± 8
percentage uncertainty	%	%

[1]

- (g) Complete the headings in the table below to record the volume of **FA 1** (aqueous sodium thiosulfate), the volume of distilled water and the volume of **FA 2** (hydrochloric acid).

In the second row copy the volumes used in **Experiment 3** from your table of results on page 4.

In the following two rows suggest volumes of each of the reagents that could be used in two further experiments, **Experiment 7** and **Experiment 8**, to investigate how the rate of reaction varies with a change in the concentration of the acid.

Do not carry out these experiments.

Experiment			
3			
7			
8			

[2]

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

Solutions **FA 3**, **FA 4**, **FA 5** and **FA 6** each contain one cation and one anion from those listed on pages 12 and 13.

- (a) Some cations interfere with tests for anions and have to be removed from the solution before the tests for anions present can be performed. One way in which this can be carried out is to precipitate the cation in the form of its insoluble carbonate.

Carry out the following tests on both **FA 3** and **FA 5**.

<i>test</i>	<i>observation</i>	
	FA 3	FA 5
To 5 cm depth of solution in a boiling tube, add all of the sodium carbonate, Na_2CO_3 , from one of the tubes provided.		
Stir the mixture.		

Retain the mixture from **FA 3** for use in (b).

[4]

- (b) Filter the mixture from **FA 3** from (a) into another boiling tube. Ignore any colour in the filtered solution.

Add 5 cm depth of dilute nitric acid. This removes any excess of carbonate ions.

Carry out the following tests on the acidified filtrate from **FA 3**.

<i>test</i>	<i>observation</i>
To 2 cm depth of the acidified filtrate from FA 3 in a test-tube, add 1 cm depth of aqueous silver nitrate, then	
add an excess of aqueous ammonia.	

[1]

- (c) Carry out the following test on **FA 4**.

<i>test</i>	<i>observation</i>
To 1 cm depth of FA 4 in a test-tube, add 1 cm depth of FA 3 , then	
add a few drops of starch solution.	

[2]

(d) Carry out the following tests.

<i>test</i>	<i>observation</i>			
	FA 3	FA 4	FA 5	FA 6
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous sodium hydroxide, then				
add a further 2 cm depth of aqueous sodium hydroxide.				
To 1 cm depth of solution in a test-tube, use a dropping pipette to add, a little at a time, 0.5 cm depth of aqueous ammonia, then				
add a further 2 cm depth of aqueous ammonia.				

[4]

(e) The results from tests in (d) should enable you to identify either a single cation in a solution, or a pair of cations which have identical reactions with the reagents used. Identify any single ion that is present or suggest a pair of ions that may be present in each of the solutions.

<i>solution</i>	<i>cation(s)</i>
FA 3	
FA 4	
FA 5	
FA 6	

[2]

- (f) Where you were unable to identify a single cation in (e), suggest a suitable reagent which would allow you to identify which cation is present in the solution.

Do not carry out this test.

The reagent to be used is

State the expected observations to identify the presence of each of the cations from the pair of ions you have given in (e).

cation 1

.....

cation 2

..... [1]

- (g) By considering the results of **all** your tests, enter one of the following responses in each of the boxes below.

- chloride
- bromide
- iodide
- no halide ion is present
- insufficient tests (have been performed to identify any halide ion)

FA 3	
FA 4	
FA 5	

[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)	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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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
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CHEMISTRY

9701/34

Advanced Practical Skills 2

October/November 2012

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.

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 13 and 14.

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	

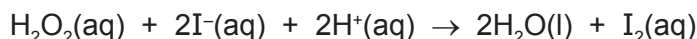
This document consists of **13** printed pages and **3** blank pages.



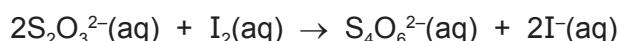
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- 1 You are to investigate how the rate of reaction between acidified hydrogen peroxide and aqueous iodide ions depends on the concentration of the hydrogen peroxide.

When hydrogen peroxide and potassium iodide are mixed in the presence of an acid, iodine, I_2 , is produced and the colour of the solution changes from colourless to a blue-black colour if starch indicator is present.



If the reaction mixture contains sodium thiosulfate, the iodine produced in the reaction above is **immediately** reduced back to iodide ions. The solution only turns blue-black when all of the sodium thiosulfate has been used up.



FB 1 is 0.23 mol dm^{-3} hydrogen peroxide, H_2O_2 .

FB 2 is 0.10 mol dm^{-3} potassium iodide, KI.

FB 3 is $0.050 \text{ mol dm}^{-3}$ sodium thiosulfate, $Na_2S_2O_3$.

FB 4 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 .

starch indicator

distilled water

Read through the instructions carefully before starting any practical work.

(a) Experiment 1

- Fill a burette with **FB 3**.
- Use the measuring cylinder labelled **A** to place 25 cm^3 of **FB 2** and 25 cm^3 of distilled water into a conical flask.
- Add to the conical flask 10.00 cm^3 of **FB 3** from the burette and 6 drops of starch indicator.
- Use the measuring cylinder labelled **B** to place 50 cm^3 of **FB 1** and 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Pour the mixture from the beaker into the conical flask and **immediately** start timing.
- Swirl the flask to ensure good mixing and place the flask on a white tile.
- Stop timing when a blue-black colour suddenly appears in the solution.
- Record, in the table on page 4, the reaction time, in seconds, to the nearest second.
- Empty, rinse and drain the conical flask.

Experiment 2

- Use the measuring cylinder labelled **A** to place 25 cm^3 of **FB 2** and 35 cm^3 of distilled water into a conical flask.
- Add to the conical flask 10.00 cm^3 of **FB 3** from the burette and 6 drops of starch indicator.
- Use the measuring cylinder labelled **B** to place 40 cm^3 of **FB 1** and 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Pour the mixture from the beaker into the conical flask and immediately start timing.
- Swirl the flask to ensure good mixing and place the flask on a white tile.
- Stop timing when a blue-black colour suddenly appears in the solution.
- Record, in the table on page 4, the reaction time, in seconds, to the nearest second.
- Empty, rinse and drain the conical flask.

Experiments 3 – 5

Carry out experiments 3 – 5 in the same way but using the volumes of solutions shown in the table.

Complete the units in the table.

Calculate all values of $\frac{(1000)}{(\text{reaction time})}$ to three significant figures.

For
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Use

Experiment	volume of FB 2 /cm ³	volume of distilled water /cm ³	volume of FB 3 /cm ³	volume of FB 1 /cm ³	volume of FB 4 /cm ³	reaction time 	$\frac{(1000)}{(\text{reaction time})}$
1	25	25	10.00	50	20		
2	25	35	10.00	40	20		
3	25	45	10.00	30	20		
4	25	55	10.00	20	20		
5	25	65	10.00	10	20		

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	

[9]

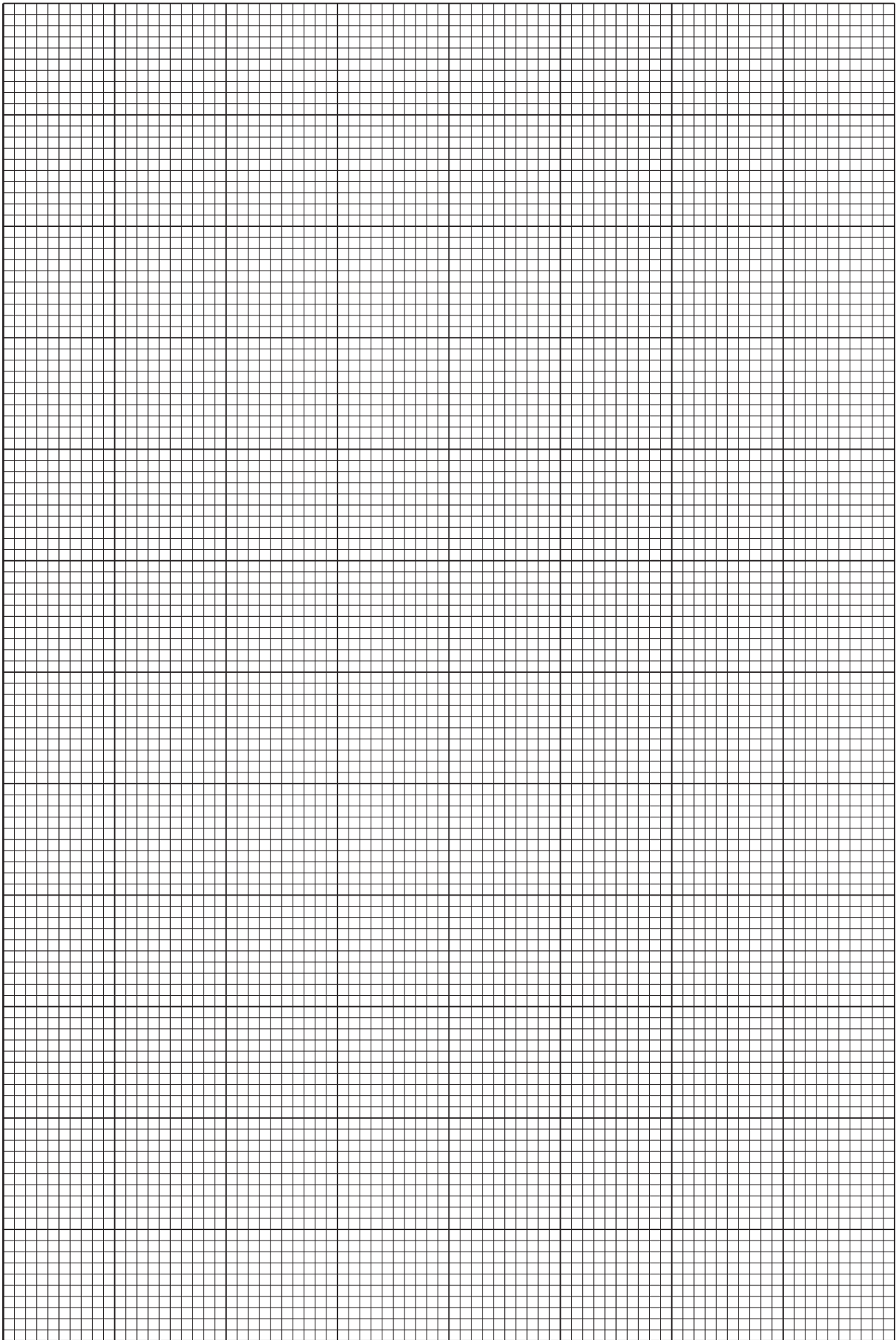
(b) The rate of reaction can be represented by the following formula.

$$\text{'rate'} = \frac{(1000)}{(\text{reaction time})}$$

On the next page plot a graph of **'rate'** against the **volume of FB 1**.

Start each of the axes at zero.

Draw the line of best fit.



[5]

I	
II	
III	
IV	
V	

(c) The concentration of hydrogen peroxide in **FB 1** is 0.23 mol dm^{-3} .

The total volume of each reaction mixture is 130 cm^3 .

(i) Calculate the initial concentration of hydrogen peroxide for each of the following experiments. Show your working.

Experiment	volume of FB 1 / cm^3	concentration of hydrogen peroxide / mol dm^{-3}
1	50	
5	10	

(ii) Use your results in (i) to show that the initial concentration of hydrogen peroxide is directly proportional to the volume of **FB 1** used in the experiment.

.....

.....

.....

.....

[3]

(d) A website states that the rate of reaction between acidified hydrogen peroxide and potassium iodide is **directly proportional** to the concentration of hydrogen peroxide.

Use your graph to decide whether the statement on the website is correct or not.

Explain your answer.

.....

.....

.....

.....

[2]

- (e) **Experiment 1** was repeated using $0.025 \text{ mol dm}^{-3}$ sodium thiosulfate instead of **FB 3**. Suggest how this would affect the reaction time. Explain your answer using the chemical equations on page 3.

.....

 [2]

- (f) Suggest a factor, other than volumes of solutions used, that could have significantly affected the rate of reaction in each of the experiments.

..... [1]

- (g) A student carrying out a similar investigation decides to repeat one of the experiments a number of times. The reaction times for these repeated experiments are listed below.

run	time / s
1	56
2	54
3	62
4	56
5	53

- (i) From these experimental results calculate an appropriate mean reaction time, correct to 1 decimal place.

mean reaction time = ... s

- (ii) Assume that the uncertainty in the mean reaction time is ± 2 seconds. Calculate this uncertainty as a percentage of the mean reaction time.

percentage uncertainty = ... %
 [2]

- (h) The experimental method you have used can be adapted to investigate how the rate of reaction would vary if the concentration of potassium iodide or the concentration of sulfuric acid were changed.

In the first line of the tables below, the volumes of **FB 2**, distilled water, **FB 3**, **FB 1** and **FB 4** used in **Experiment 2** are recorded.

Complete the following table, suggesting volumes for each of the reagents that could be used in a further experiment to investigate how the rate of reaction varies with a change in the volume of **potassium iodide, FB 2**.

Do not carry out this experiment.

Experiment	volume of FB 2	volume of distilled water	volume of FB 3	volume of FB 1	volume of FB 4
	/cm ³	/cm ³	/cm ³	/cm ³	/cm ³
2	25	35	10.00	40	20

Complete the following table, suggesting volumes for each of the reagents that could be used in a further experiment to investigate how the rate of reaction varies with a change in the volume of **sulfuric acid, FB 4**.

Do not carry out this experiment.

Experiment	volume of FB 2	volume of distilled water	volume of FB 3	volume of FB 1	volume of FB 4
	/cm ³	/cm ³	/cm ³	/cm ³	/cm ³
2	25	35	10.00	40	20

[1]

[Total: 25]

2 Qualitative Analysis

For
Examiner's
Use

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.

Solutions **FB 5**, **FB 6** and **FB 7** each contain one cation and one anion from those listed on pages 13 and 14.

Half fill a 250 cm³ beaker with water. Heat the beaker and its contents to boiling then stop heating. You will need this as a hot water bath in **(f)**.

(a) Carry out the following tests on **FB 5**, **FB 6** and **FB 7** using aqueous sodium hydroxide.

- To 1 cm depth of **FB 5**, **FB 6** and **FB 7** in separate boiling tubes add 1 cm depth of aqueous sodium hydroxide.
- Shake the tube to mix the solutions then add a further 2 cm depth of aqueous sodium hydroxide.
- If **no** precipitate has formed in a solution for either of the previous steps, carefully **warm** the boiling tube and its contents.
Care: if solutions containing sodium hydroxide are heated too strongly they may be ejected from the tube.

Record your results in an appropriate form in the space below.

I	
II	
III	
IV	

[4]

(b) Carry out the following tests on **FB 5**, **FB 6** and **FB 7** using aqueous ammonia.

- To 1 cm depth of **FB 5**, **FB 6** and **FB 7** in separate test-tubes add 1 cm depth of aqueous ammonia.
- Shake the tube to mix the solutions then add a further 2 cm depth of aqueous ammonia.
- Record your results in an appropriate form in the space below.

[2]

(c) From your observations in (a) and (b), identify the cation present in each of the following solutions.

<i>solution</i>	<i>cation</i>
FB 5	
FB 6	
FB 7	

[1]

(d) Each of the solutions **FB 5**, **FB 6** and **FB 7** contains either a sulfate or a sulfite anion.

- (i) Which single reagent, when added to the solution, could confirm that either a sulfate or a sulfite is present?

.....

Which additional reagent, when added to the same test-tube, would identify which of these two ions is present?

.....

- (ii) Carry out the tests on **FB 5**, **FB 6** and **FB 7** using the reagents you have selected and record your observations in the table below.

	<i>observation</i>		
	FB 5	FB 6	FB 7
reagent used:			
followed by:			

- (iii) Identify the anion present in each solution. Explain your answer.

.....

.....

.....

[4]

- (e) Carry out the following test.

<i>test</i>	<i>observation</i>
To 1 cm depth of FB 5 in a boiling tube, add 2 cm depth of the aqueous hydrogen peroxide, FB 9 . Warm the tube, then,	
add 2 cm depth of aqueous sodium hydroxide.	

[2]

(f) Divide the sample of solid **FB 8** in two. Use one portion in each of the following tests.

Test 1

- Place the portion of **FB 8** in a boiling tube.
- Add 2 cm depth of dilute sulfuric acid.
- Warm with a Bunsen burner.
- Test any vapour evolved with litmus paper.

Observation

.....

Test 2

- Reheat the water bath to boiling, then turn off the Bunsen burner.
- Place the remaining **FB 8** in a dry test-tube.
- Add 2 cm depth of ethanol.
- Use a dropping pipette to add 2–3 drops of concentrated sulfuric acid.
Care – concentrated sulfuric acid is very corrosive.
- Warm the tube in the hot-water bath.
- After 3–4 minutes of warming tip the contents of the test-tube into a 100 cm³ beaker, $\frac{3}{4}$ full of cold water.
- Cautiously smell the contents of the beaker.

Observation

.....

Use your observations above to suggest the type of compound present in **FB 8**.

.....

[2]

[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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CHEMISTRY

9701/35

Advanced Practical Skills 1

October/November 2012

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.
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Do not use staples, paper clips, highlighters, glue or correction fluid.
DO **NOT** WRITE IN ANY BARCODES.

Answer **all** questions.

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 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

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1	
2	
3	
Total	

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



- 1 You are to investigate the temperature change when a piece of magnesium ribbon is added to hydrochloric acid. You will measure the temperature at regular intervals during the reaction.

FA 1 is approximately 2 mol dm^{-3} hydrochloric acid, HCl .
You are also provided with magnesium ribbon.

Read through the instructions carefully before starting any practical work.

(a) Method

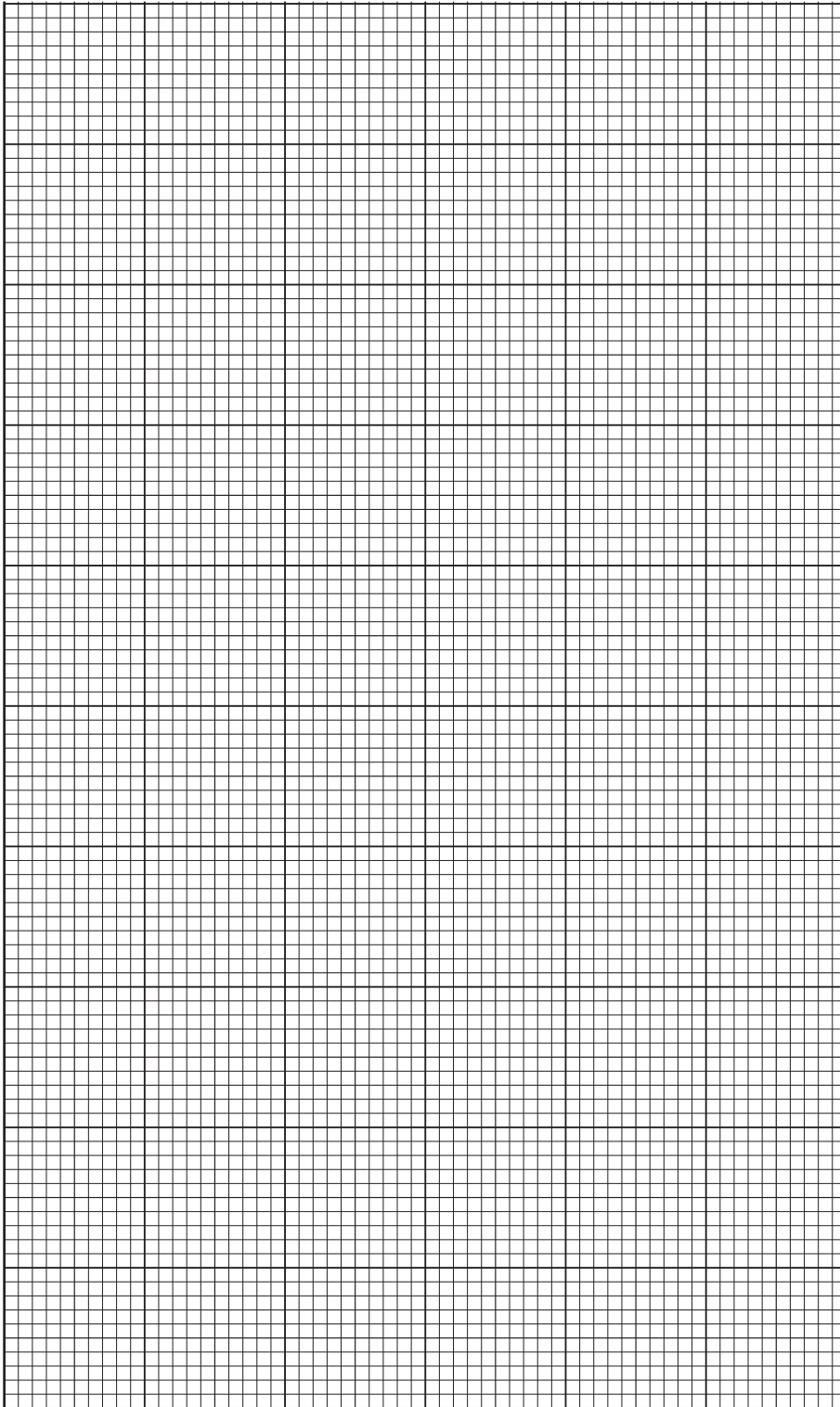
- Curl or fold the magnesium ribbon so that it will just fit in the bottom of the plastic cup. **Remove the magnesium from the cup.**
- Support the plastic cup in a 250 cm^3 beaker.
- Use a measuring cylinder to transfer 50 cm^3 of **FA 1** into the **empty** plastic cup.
- Measure and record in the table below, the initial temperature of the acid in the cup.
- Start the stop watch. Measure and record the temperature of **FA 1** in the cup after 1 minute, 2 minutes and 3 minutes.
- At time $3\frac{1}{2}$ minutes, add the curled or folded magnesium to the **FA 1** in the cup and stir the mixture. Make sure the magnesium is submerged in the acid.
- From time 4 minutes, continue to 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
temperature/°C											

[1]

- (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 between time 5 minutes and 10 minutes.
- 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) In the experiment you have just carried out, explain how you know that the hydrochloric acid was in excess.

.....

- (iii) 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?

maximum error = ... °C

Calculate the maximum percentage error when measuring a temperature **rise** of 7.5 °C.

maximum percentage error = ... %

- (iv) Apart from errors associated with the thermometer, suggest one significant source of error in the procedure used in this experiment. Suggest an improvement that could be made to reduce this error.

.....

- (v) The experiment was repeated on another day when the temperature of the room was much higher than when the original experiment was carried out.

Discuss the effect of this higher room temperature on the results of the experiment.

.....

[7]

[Total: 12]

- 2 The concentration of the acid, **FA 2**, can be found by titrating it against aqueous sodium carbonate of known concentration.

FA 2 is hydrochloric acid, HCl .

FA 3 is sodium carbonate, Na_2CO_3 .
methyl orange indicator

You are to determine the concentration of **FA 2**.

(a) Method

- Weigh a 100 cm^3 beaker.
- Weigh out between 1.3 g and 1.5 g of **FA 3** into this beaker.
- Record the weighings and the mass of **FA 3** added.

mass of **FA 3** = ... g

- Add about 50 cm^3 of distilled water to the beaker.
- Stir with a glass rod until all the solid has dissolved.
- Pour the solution from the beaker into the 250 cm^3 graduated (volumetric) flask.
- Wash the beaker with distilled water and add the washings to the flask.
- Make up the contents of the graduated flask to the 250 cm^3 mark with distilled water.
- Invert the flask as many times as you think necessary to mix the solution of **FA 3** thoroughly.
- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of the solution of **FA 3** into a conical flask.
- Add methyl orange indicator.
- Titrate the solution of **FA 3** with **FA 2**.
- 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 2** added in each accurate titration.

For
Examiner's
Use

I	
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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 2** [1]

(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 sodium carbonate were present in 25.0 cm³ of the solution you pipetted for the titration.
[A_r: C, 12.0; O, 16.0; Na, 23.0]

moles of Na₂CO₃ = ... mol

- (ii) The equation for the reaction between hydrochloric acid and sodium carbonate is shown below.



Use your answers to (b) and (c)(i) to calculate the concentration, in mol dm⁻³, of the hydrochloric acid, **FA 2**.

concentration of HCl, **FA 2** = ... mol dm⁻³

- (iii) **FA 2** is hydrochloric acid, HCl , made using 50.0 cm^3 of **FA 1** diluted to 1.00 dm^3 with distilled water.
Calculate the concentration, in mol dm^{-3} , of the hydrochloric acid, **FA 1**.

For
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Use

concentration of HCl , **FA 1** = ... mol dm^{-3}
[5]

[Total: 13]

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 solutions **FA 4**, **FA 5** and **FA 6** and solid **FA 7**. Each of these contain a transition metal ion.

(i) Carry out the following tests on **FA 4**.

<i>test</i>	<i>observations</i>
To 1 cm depth of FA 4 in a test-tube, add an equal depth of concentrated hydrochloric acid with care .	
To 1 cm depth of FA 4 in a test-tube, add aqueous ammonia.	
To 1 cm depth of FA 4 in a test-tube, add aqueous silver nitrate.	
To 1 cm depth of FA 4 in a test-tube, add an equal depth of aqueous potassium iodide then, add a few drops of starch solution.	

- (ii) From these tests identify **FA 4**.

.....

- (iii) Suggest what happened to the potassium iodide in its reaction with **FA 4**. Use your practical results to explain your answer.

.....

.....

[6]

- (b) **FA 5**, **FA 6** and **FA 7** each contain compounds of the same transition element.

- (i) Complete the following table.

<i>test</i>	<i>observations</i>
To 1 cm depth of aqueous iron(II) sulfate in a test-tube, add a few drops of FA 5 .	
To 1 cm depth of FA 6 in a test-tube, add an equal depth of aqueous sodium hydroxide then,	
add 1 cm depth of hydrogen peroxide.	
To 1 cm depth of FA 6 in a test-tube, add aqueous barium chloride or aqueous barium nitrate then,	
add an excess of either hydrochloric acid or nitric acid.	
To 1 cm depth of hydrogen peroxide in a boiling tube , add a small spatula measure of FA 7 .	

- (ii) From these tests identify **FA 6**. Explain how your observations support this conclusion.

identity

evidence for cation

.....

evidence for anion

.....

- (iii) It was suggested that Fe^{2+} was oxidised when iron(II) sulfate reacted with **FA 5**.

To 1 cm depth of aqueous iron(II) sulfate in a test-tube add **FA 5** dropwise until a **pale** permanent pink colour persists.

Devise and carry out a test to show that the Fe^{2+} ions in this solution have been oxidised.

Record the test used and the result obtained in the space below.

[9]

[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 $\text{NO} \rightarrow$ (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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UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

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CHEMISTRY

9701/36

Advanced Practical Skills 2

October/November 2012

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.

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 15 and 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

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **13** printed pages and **3** blank pages.



- 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]

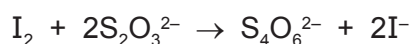
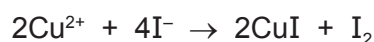
I	
II	
III	
IV	
V	
VI	

- (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, $\text{S}_2\text{O}_3^{2-}$, present in the volume of **FB 2** in (b).

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

- (ii) Using the equations above, deduce the number of moles of Cu^{2+} ions present in each 25.0 cm³.

moles of Cu^{2+} = ... mol

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

concentration of CuSO_4 = ... 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

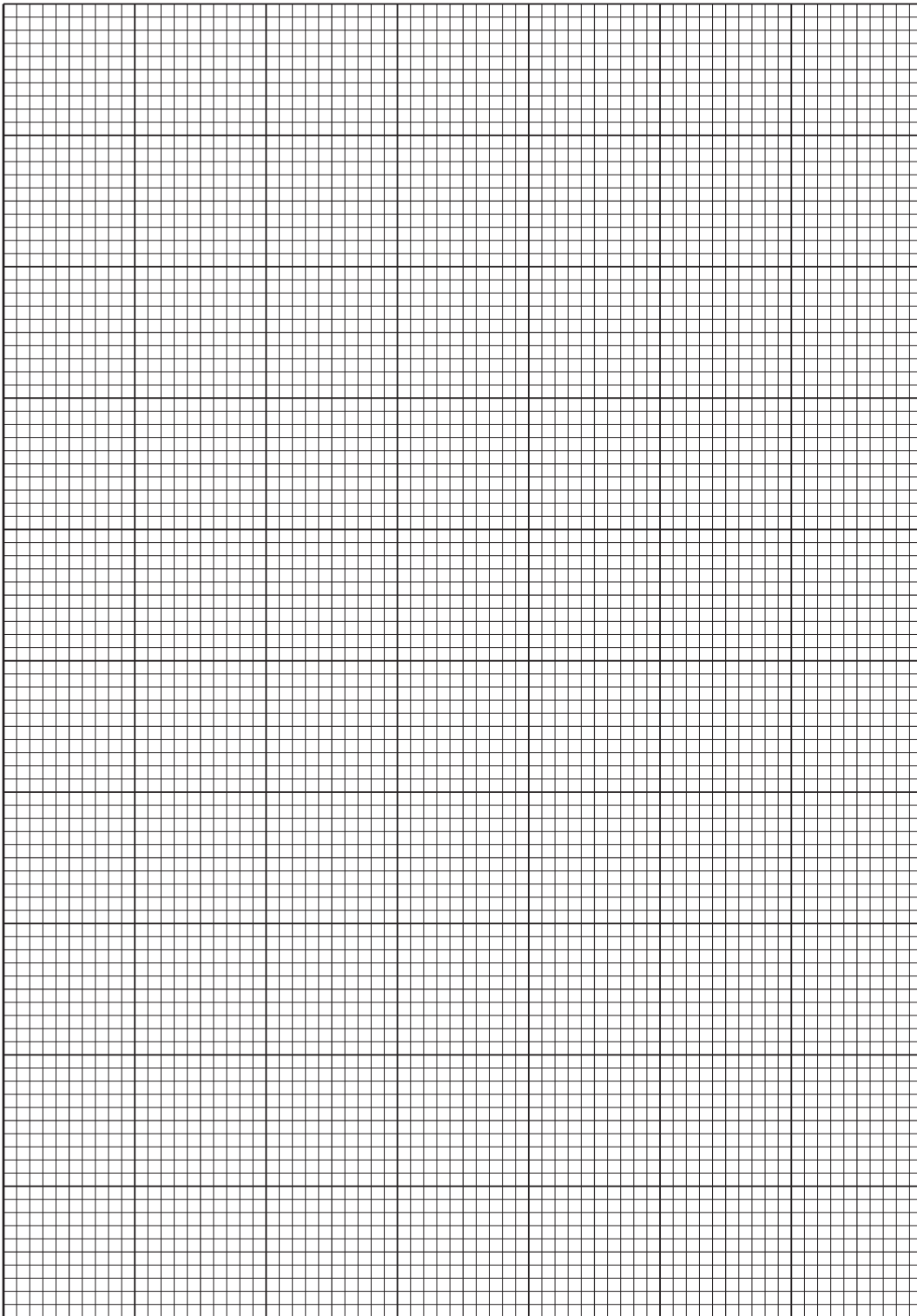
- Support the plastic cup in a 250 cm³ beaker.
- Use a measuring cylinder to transfer 50 cm³ of **FB 4** into the plastic cup.
- Measure and record in the table below, the initial temperature of **FB 4** in the cup.
- Start the stop watch. Measure and record the temperature of **FB 4** in the cup after 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 your 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 ?

maximum error = ... $^{\circ}\text{C}$

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

maximum percentage error = ... % [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]

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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