



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

May/June 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 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.

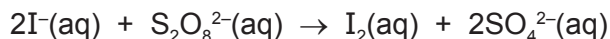
Session
Laboratory

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Total	

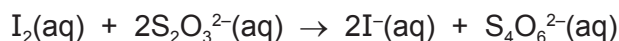
This document consists of **12** printed pages.



- 1 When iodide ions are mixed with peroxodisulfate ions, $\text{S}_2\text{O}_8^{2-}$, iodine is formed.



The rate of this reaction can be measured by adding thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, and some starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.



When all the thiosulfate ions have reacted, the iodine which continues to be produced then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the reaction mixture to turn blue-black.

You are to investigate how the rate of reaction is affected by changing the concentration of the peroxodisulfate ions.

FA 1 is $0.0200 \text{ mol dm}^{-3}$ aqueous potassium peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$.

FA 2 is 1.00 mol dm^{-3} aqueous potassium iodide, KI .

FA 3 is $0.00500 \text{ mol dm}^{-3}$ aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.
starch indicator

Read through the instructions carefully and prepare a table for your results on page 3 before starting any practical work.

(a) Method

Experiment 1

- Fill the burette labelled **FA 1** with aqueous potassium peroxodisulfate, **FA 1**.
- Run 20.00 cm^3 of **FA 1** into a 100 cm^3 beaker.
- Using a 25 cm^3 measuring cylinder add the following to a second 100 cm^3 beaker:
 - 20 cm^3 of **FA 2**
 - 10 cm^3 of **FA 3**
- Add 10 drops of starch indicator to the second beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

Experiment 2

- Fill a second burette with distilled water.
- Run 10.00 cm^3 of **FA 1** into the first 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into the beaker containing **FA 1**.
- Using a 25 cm^3 measuring cylinder add the following to the second 100 cm^3 beaker:
 - 20 cm^3 of **FA 2**
 - 10 cm^3 of **FA 3**
- Add 10 drops of starch indicator to the second beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

Experiments 3 – 5

- Carry out three further experiments to investigate how the reaction time changes with different volumes of potassium peroxodisulfate.
Remember that the combined volume of **FA 1** and distilled water must always be 20.00 cm³.
Do not use a volume of **FA 1** that is less than 6.00 cm³.

Record all your results in a single table. You should include the volume of potassium peroxodisulfate, the volume of distilled water and the reaction time.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	

[9]

- (b) In order to convert the times measured in the experiments into rates of reaction, it is necessary first to work out the concentration of I₂(aq) that would have been produced in the reaction time if the thiosulfate had not been present. You must show your working.

- (i) Calculate how many moles of thiosulfate ions, S₂O₃²⁻, were added in each experiment.

moles of S₂O₃²⁻ = ... mol

- (ii) Calculate how many moles of iodine, I₂, must have been produced to react with this amount of thiosulfate ions.

moles of I₂ = ... mol

- (iii) Calculate the concentration of iodine from (ii) in the total reaction volume.

concentration of I₂ = ... mol dm⁻³
[3]

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

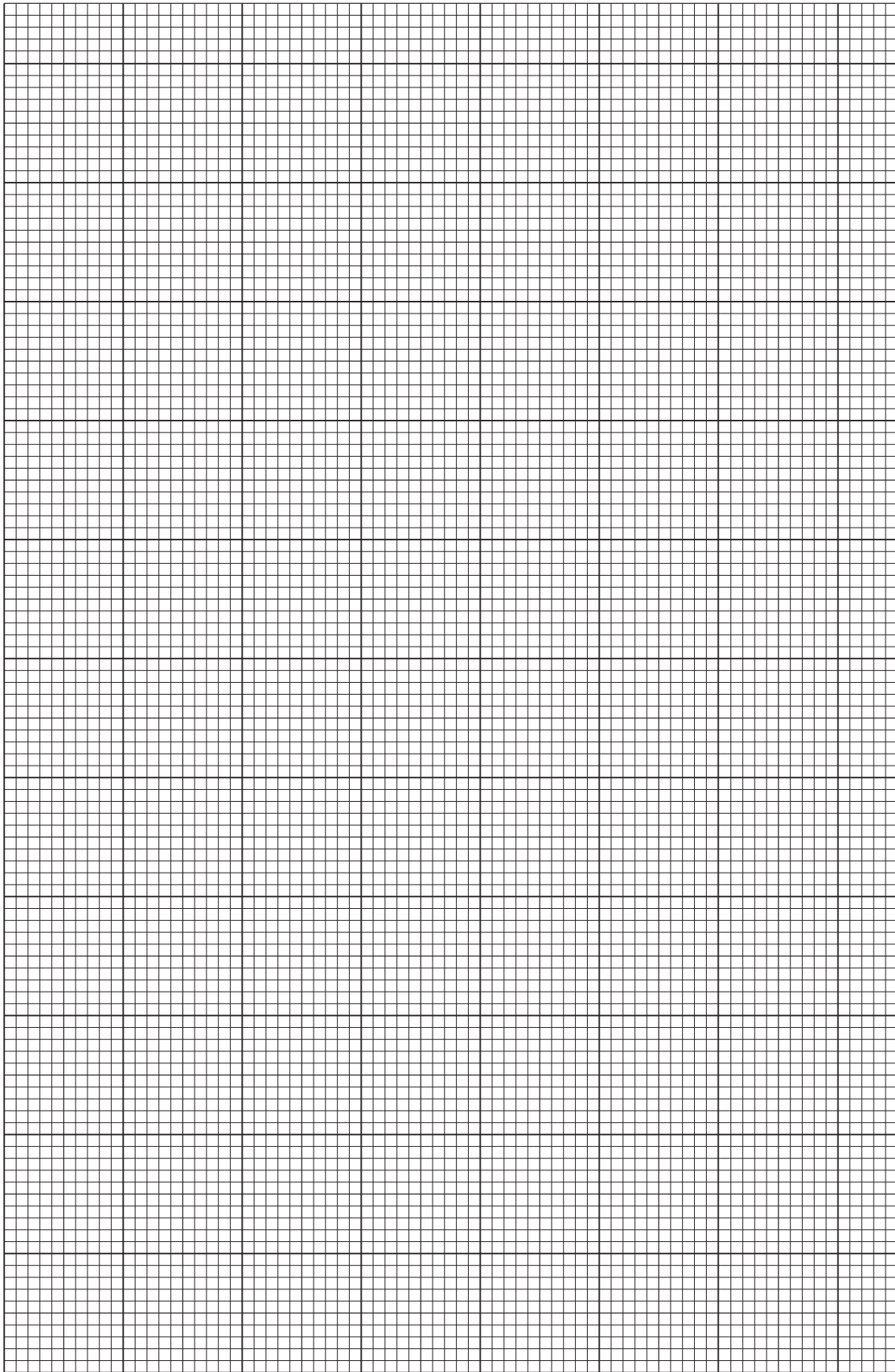
$$\text{'rate'} = \frac{\text{concentration of I}_2 \text{ from (b)(iii)}}{\text{reaction time}} \times 10^6$$

Use your experimental results to complete the following table. Include the volume of **FA 1**, the reaction time and 'rate' with their units.

If you were unable to answer (b)(iii), you may assume that the concentration of iodine is $4.25 \times 10^{-4} \text{ mol dm}^{-3}$ (This is not the correct value).

[2]

(d) On the grid opposite, plot the 'rate' against the volume of **FA 1**. Draw a line of best fit.



I	
II	
III	
IV	

[4]

- (e) In your experiments, the volume of **FA 1** represents the concentration of potassium peroxodisulfate. From your results, what conclusion can you draw about the relationship between the rate of reaction and the concentration of potassium peroxodisulfate?

.....

 [2]

- (f) Assume that the error in the time measured for each reaction was ± 0.5 s.

- (i) Calculate the maximum percentage error in the reaction time you recorded in **Experiment 1**.

maximum percentage error = ...%

- (ii) Assuming this is the **only** source of error, calculate the minimum reaction rate for **Experiment 1**.

minimum reaction rate = ...

- (iii) Suggest an additional source of error in these experiments and what improvement could be made to reduce this error.

.....

[4]

I	
II	
III	
IV	

- (g) (i) Carry out one additional experiment using the following volumes of each reagent. Use the same method as in (a), mix **FA 2**, **FA 3**, the distilled water and the starch together and start the reaction by adding **FA 1** to this mixture.

- 10.00 cm³ of **FA 1**
- 20 cm³ of **FA 2**
- 20 cm³ of **FA 3**
- 10 drops of starch

Record the time for the reaction to go blue-black.

- (ii) Explain the relationship between this time and the one you recorded in **Experiment 2**.

.....

.....

.....

[2]

[Total: 26]

2 Qualitative Analysis

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

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

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

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

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

No additional tests for ions present should be attempted.

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

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

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

(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 1 cm depth of each solution in a test-tube and record your observations in the following table. Test the solubility of any precipitate you observe in an excess of each reagent.

*For
Examiner's
Use*

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

[5]

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

ion	Cu^{2+}	Pb^{2+}	OH^-	CrO_4^{2-}	Cl^-
solution					

[3]

I	
II	
III	
IV	
V	

I	
II	
III	

(c) The anion in **FA 9** is either the sulfite ion, SO_3^{2-} , or the sulfate ion SO_4^{2-} .

- (i) Describe a single test you could carry out that would give positive results for both of these ions.

.....

.....

- (ii) Describe a test you could carry out that would distinguish between these two ions.

.....

.....

- (iii) Carry out both of these tests and record your results in an appropriate form in the space below.

- (iv) Which anion is present in **FA 9**?

.....

I	
II	
III	
IV	
V	
VI	

[6]

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

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NAME

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CANDIDATE
NUMBER

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CHEMISTRY

9701/32

Advanced Practical Skills 2

May/June 2012

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

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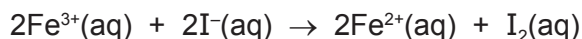
Session
Laboratory

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Total	

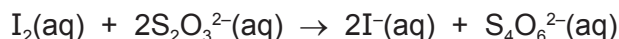
This document consists of **12** printed pages.



- 1 Iodide ions are oxidised by iron(III) ions in the presence of acid.



The rate of this reaction can be measured by adding thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, and some starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.



When all the thiosulfate ions have reacted, the iodine which continues to be produced then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the reaction mixture to turn blue-black.

You are to investigate how the rate of reaction is affected by changing the concentration of the reagents.

FB 1 is $0.0500 \text{ mol dm}^{-3}$ aqueous acidified iron(III) chloride, FeCl_3 .

FB 2 is $0.0500 \text{ mol dm}^{-3}$ aqueous potassium iodide, KI .

FB 3 is $0.00500 \text{ mol dm}^{-3}$ aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FB 4 is starch indicator.

Read through the instructions carefully and prepare a table for your results on page 3 before starting any practical work.

(a) Method

Experiment 1

- Using a 25 cm^3 measuring cylinder add the following to a 100 cm^3 beaker:
 - 10 cm^3 of **FB 2**
 - 20 cm^3 of **FB 3**
 - 10 cm^3 of **FB 4**
- Fill the burette labelled **FB 1** with aqueous acidified iron(III) chloride, **FB 1**.
- Run 20.00 cm^3 of **FB 1** into a second 100 cm^3 beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

Experiment 2

- Using a 25 cm^3 measuring cylinder add the following to a 100 cm^3 beaker:
 - 10 cm^3 of **FB 2**
 - 20 cm^3 of **FB 3**
 - 10 cm^3 of **FB 4**
- Fill a second burette with distilled water.
- Run 10.00 cm^3 of **FB 1** into a second 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into the beaker containing **FB 1**.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

Experiments 3 – 5

- Carry out three further experiments to investigate how the reaction time changes with different volumes of iron(III) chloride.
Remember that the combined volume of **FB 1** and distilled water must always be 20.00 cm³.
Do not use a volume of **FB 1** that is less than 6.00 cm³.

Record all your results in a single table. You should include the volume of iron(III) chloride, the volume of distilled water and the reaction time.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	

[9]

- (b) In order to convert the times measured in the experiments into rates of reaction, it is necessary first to work out the change in the iron(III) ion concentration that occurs from the time when the mixtures are combined to the time when the solution turns blue-black. You must show your working.

- (i) Calculate how many moles of thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, are added in each experiment.

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

- (ii) Calculate how many moles of iodine, I_2 , reacted with the number of moles of thiosulfate ions calculated in (i).

moles of I_2 = ... mol

- (iii) Calculate how many moles of iron(III) ions, Fe^{3+} , reacted to form the number of moles of iodine calculated in (ii).

moles of Fe^{3+} = ... mol

- (iv) Use your answer from (iii) to calculate the decrease in concentration of iron(III) ions in the total reaction mixture (60 cm³) from the start of the experiment to the point when the solution turned blue-black.

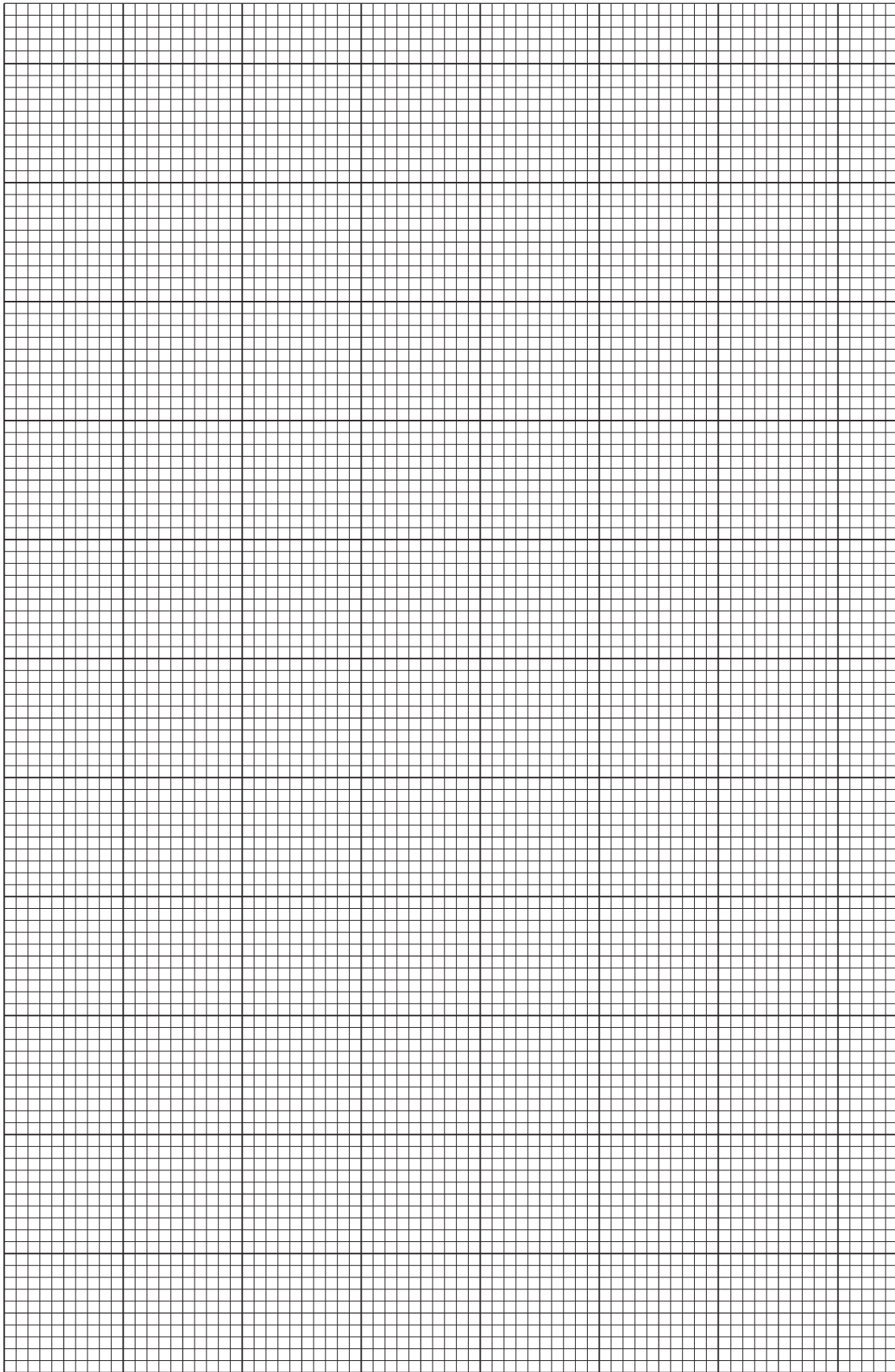
decrease in Fe^{3+} concentration = ... mol dm⁻³

[3]

$$\text{'rate'} = \frac{\text{decrease in Fe}^{3+} \text{ concentration from (b)(iv)}}{\text{reaction time}} \times 10^6$$

If you were unable to answer **(b)(iv)**, you may assume that the decrease in Fe^{3+} concentration is $2.25 \times 10^{-3} \text{ mol dm}^{-3}$ (This is not the correct value).

(d) On the grid opposite, plot 'rate' against the volume of **FB 1**. Draw a line of best fit.



I	
II	
III	
IV	

[4]

- (e) In your experiments, the volume of **FB 1** represents the concentration of iron(III) chloride. From your results, what conclusion can you draw about the relationship between the rate of reaction and the concentration of iron(III) chloride?

.....

 [2]

- (f) One source of error in this experiment arises from measuring the volumes of solutions.

- (i) Calculate the maximum percentage error in the volume of **FB 1** used in **Experiment 1**.

maximum percentage error = ...%

- (ii) Other than errors involving measurements of volumes, suggest an additional source of error in these experiments.

.....

- (iii) In this experiment, thiosulfate ions reduce iodine while iron(III) ions oxidise iodide ions. What other reaction might take place that would affect your confidence in the conclusions you made in (e)?

.....

[4]

I	
II	
III	
IV	

- (g) (i) Carry out one additional experiment using the following volumes of each reagent. Use the same method as in (a), mix **FB 2**, **FB 3** and **FB 4** together and start the reaction by adding this mixture to **FB 1** and the distilled water.

- 10.00 cm³ of **FB 1**
- 20.00 cm³ of distilled water
- 10 cm³ of **FB 2**
- 10 cm³ of **FB 3**
- 10 cm³ of **FB 4**

Record the time for the reaction to go blue-black.

- (ii) Explain the relationship between this time and the one you recorded in **Experiment 2**.

.....

.....

.....

[2]

[Total: 26]

2 Qualitative Analysis

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

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

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

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

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

No additional tests for ions present should be attempted.

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

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

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

(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.

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

*For
Examiner's
Use*

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

[5]

I	
II	
III	
IV	
V	

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

ion	Pb ²⁺	Ba ²⁺	H ⁺	CrO ₄ ²⁻	SO ₄ ²⁻	Cl ⁻
solution						

[3]

I	
II	
III	

(c) The anion in **FB 9** is either the nitrite ion, NO_2^- , or the nitrate ion, NO_3^- .

- (i) Describe a test you could carry out that would give positive results for both of these ions.

.....

.....

- (ii) Describe a test you could carry out that would distinguish between these two ions.

.....

.....

- (iii) Carry out both of these tests and record your results in an appropriate form in the space below.

- (iv) Which anion is present in **FB 9**?

.....

[6]

[Total: 14]

I	
II	
III	
IV	
V	
VI	

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
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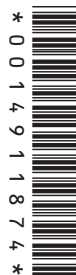
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CHEMISTRY

9701/33

Advanced Practical Skills 1

May/June 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 11 and 12.

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

Session
Laboratory

For Examiner's Use	
1	
2	
Total	

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



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

This experiment involves three steps.

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

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

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

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

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

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

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

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

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

Read through the whole method before starting any practical work.

(a) Method

Step 1

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

Step 2

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

The rough titre is cm^3 .

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

Step 3

- Stir the mixture from **Step 1** again to ensure that all the solid has dissolved.
- Transfer the solution to the 250 cm³ graduated (volumetric) flask labelled **FA 5**.
- Rinse the beaker twice with a little distilled water and add the washings to the graduated flask.
- Make the solution up to 250 cm³ with distilled water. Ensure that the contents of the flask are thoroughly mixed.
- Transfer 25.0 cm³ of this solution, **FA 5**, into a second conical flask using a second pipette.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm³.

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

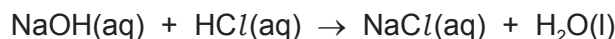
I	
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VIII	
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X	
XI	
XII	

[12]

Calculations

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

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



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

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

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

moles of NaOH = ... mol

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

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

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

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

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

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

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

moles of NaOH = ... mol

- (iii) Use your answer from (ii) to determine the number of moles of hydrochloric acid in 25.0 cm^3 of **FA 5**.

moles of HCl in 25.0 cm^3 of **FA 5** = ... mol

- (iv) Use your answer from (iii) to determine the number of moles of hydrochloric acid in 250 cm^3 of **FA 5**.

moles of HCl in 250 cm^3 of **FA 5** = ... mol
[4]

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

.....

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

moles of HCl = (b)(iv) – (c)(iv)

= mol

- (iii) Use your answers from (i) and (ii) to calculate the mass of CaCO_3 in **FA 4**.
[A_r: C, 12.0; O, 16.0; Ca, 40.1]
(If you were unable to answer (d)(ii), you may assume that the number of moles of hydrochloric acid that reacted with calcium carbonate was 0.0351 mol .)

mass of CaCO_3 in **FA 4** = ... g

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

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

For
Examiner's
Use

I	
II	
III	
IV	

I	
II	
III	
IV	

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

maximum error in a burette reading = ... cm³

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

maximum percentage error = ... %
[2]

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

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

[Total: 25]

2 Qualitative analysis

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

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

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

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

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

No additional tests for ions present should be attempted.

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

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

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

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

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

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

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

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VI	

[6]

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

The transition metal present in the three compounds is

Explain how your observations support your conclusion.

.....

.....

.....

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

.....

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

.....

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

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

.....

[3]

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

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

The first reagent is

and this is followed by

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

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

FA 9 contains

FA 10 contains

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

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

Do not carry out these experiments.

expected observation with **FA 9**

.....

expected observation with **FA 10**

.....

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

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

observation

.....

[6]

[Total: 15]

I	
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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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CHEMISTRY

9701/34

Advanced Practical Skills 2

May/June 2012

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



- 1 You are to determine the percentage by mass of sodium ethanedioate in a mixture of sodium ethanedioate and ethanedioic acid.

This experiment involves two steps.

In step one, you will carry out a titration to find the amount of acid, $\text{C}_2\text{O}_4\text{H}_2$, present in **FB 3**.

In step two, you will carry out a second titration to find the total amount of ethanedioate ion, $\text{C}_2\text{O}_4^{2-}$, present in **FB 3**.

Finally, you will use the values found in the two steps to calculate the percentage by mass of sodium ethanedioate in **FB 3**.

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

FB 2 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FB 3 is a mixture of aqueous sodium ethanedioate, $\text{C}_2\text{O}_4\text{Na}_2$, and ethanedioic acid, $\text{C}_2\text{O}_4\text{H}_2$.

FB 4 is approximately 2 mol dm^{-3} sulfuric acid.
phenolphthalein indicator

Read through the whole method before starting any practical work.

(a) Method

Step 1

- Fill the burette labelled **FB 1** with **FB 1**.
- Pipette 25.0 cm^3 of **FB 3** into a conical flask.
- Add about three drops of phenolphthalein.
- 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 1** added in each accurate titration.

Step 2

- Pipette 25.0 cm³ of **FB 3** into a conical flask.
- Using a measuring cylinder, add about 25 cm³ of 2 mol dm⁻³ sulfuric acid, **FB 4**, to the flask.
- Place the conical flask on a tripod and gauze and heat to about 80 °C.
- Fill the burette labelled **FB 2** with **FB 2**.
- Use an appropriate method to carefully transfer the hot conical flask onto a white tile under the burette.
- Titrate the mixture in the conical flask with **FB 2** until a permanent pale pink colour is seen. If a permanent brown colour is seen, stop the titration and begin **Step 2** again.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is cm³.

- 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.

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[12]

Calculations

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

- (b) (i) From your accurate titration results in **Step 1**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

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

- (ii) Write an equation for the reaction between sodium hydroxide and ethanedioic acid to give sodium ethanedioate and water.
-

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

moles of NaOH = ... mol

- (iv) Use your answer to (iii) to determine the number of moles of ethanedioic acid in 25.0 cm³ of **FB 3**.

moles of C₂O₄H₂ in 25.0 cm³ of **FB 3** = ... mol
[2]

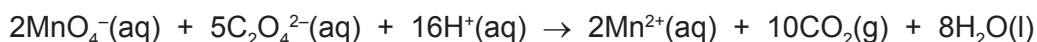
- (c) (i) From your accurate titration results in **Step 2**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm³ of **FB 3** required cm³ of **FB 2**.

- (ii) Use your answer from (i) to calculate the number of moles of potassium manganate(VII), **FB 2**, required to react with 25.0 cm³ of **FB 3** in **Step 2**.

moles of KMnO₄ = ... mol

- (iii) The equation for the reaction between acidified manganate(VII) ions and ethanedioate ions is shown below.



Calculate the total number of moles of ethanedioate ions in 25.0 cm³ of **FB 3**.

total moles of C₂O₄²⁻ in 25.0 cm³ of **FB 3** = ... mol

- (iv) Use your answers to (b)(iv) and (c)(iii) to calculate the number of moles of ethanedioate ions which came from the sodium ethanedioate dissolved in 25.0 cm³ of **FB 3**.

moles of C₂O₄²⁻ from C₂O₄Na₂ in 25.0 cm³ of **FB 3** = ... mol
[4]

- (d) (i) Use your answer to (b)(iv) to calculate the mass of ethanedioic acid, C₂O₄H₂, in 25.0 cm³ of **FB 3**.

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

(If you were unable to answer (b)(iv), you may assume that the number of moles of ethanedioic acid is 6.51 × 10⁻⁴ mol.)

mass of ethanedioic acid = ... g

- (ii) Use your answer to (c)(iv) to calculate the mass of sodium ethanedioate, C₂O₄Na₂ in 25.0 cm³ of **FB 3**.

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

(If you were unable to answer (c)(iv), you may assume that the number of moles of sodium ethanedioate is 4.13 × 10⁻⁴ mol.)

mass of sodium ethanedioate = ... g

- (iii) Calculate the percentage by mass of sodium ethanedioate present in **FB 3**.

Percentage by mass of sodium ethanedioate present is %
[4]

For
Examiner's
Use

I	
II	
III	
IV	

I	
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IV	

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

maximum error = ... cm^3

- (ii) A student suggested that using a burette to measure the 25.0 cm^3 of acid would give a more accurate result than using a pipette. The percentage error of a 25.0 cm^3 pipette is 0.24 %. Is the student correct? Explain your answer.

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

[2]

- (f) A student decided to use a 25.0 cm^3 pipette instead of a measuring cylinder to measure the volume of **FB 4** in **Step 2**.

State and explain whether this alteration will improve the accuracy of the calculation of the percentage by mass of sodium ethanedioate in the mixture.

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

[Total: 25]

2 Qualitative analysis

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

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

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

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

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

No additional tests for ions present should be attempted.

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

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

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

- (a) Compounds **FB 5**, **FB 6** and **FB 7** contain the same non-metal but in three different oxidation states. You are provided with solid samples of **FB 5**, **FB 6** and **FB 7**. Carry out the tests described below and record your observations in the table.

<i>test</i>	<i>observations</i>
(i) To 1 cm depth of dilute hydrochloric acid in a test-tube add a small spatula measure of FB 5 .	
(ii) To 1 cm depth of dilute sulfuric acid in a boiling tube add the same depth of aqueous potassium iodide. Add a small spatula measure of FB 5 .	
(iii) To 1 cm depth of dilute sulfuric acid in a test-tube add about ten drops of aqueous potassium manganate(VII). Add a small spatula measure of FB 5 .	

<i>test</i>	<i>observations</i>
(iv) Place a small spatula measure of FB 6 into a hard glass test-tube. Heat the contents gently.	
(v) Place a small spatula measure of FB 6 into a boiling tube. Dissolve the solid in 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture with care .	
(vi) Place a small spatula measure of FB 7 into a hard glass test-tube. Heat the contents gently at first, then heat more strongly. Allow to stand for a few minutes	
(vii) Place a small spatula measure of FB 7 into a boiling tube. Dissolve the solid in about 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture with care .	

[6]

I	
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VI	

- (b) (i) From your observations in (a), identify the non-metal present in **FB 5**, **FB 6** and **FB 7**.

.....

- (ii) Suggest the oxidation state of the non-metal in **FB 5** and **FB 6**.

The oxidation state of the non-metal in **FB 5** is

The oxidation state of the non-metal in **FB 6** is

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

.....

[3]

(c) Solid compounds containing Fe^{2+} and Ni^{2+} are usually green. One of these ions is present in **FB 8** and the other in **FB 9**. Both **FB8** and **FB9** are aqueous solutions.

- (i) Use the Qualitative Analysis Notes on page 10 to select two reagents that, **used in separate tests**, could identify the presence of the Fe^{2+} ion.

The reagents are

and

- (ii) Use your chosen reagents to carry out tests on **both FB 8** and **FB 9**.
Record your results in an appropriate form in the space below.

- (iii) From the results of the tests in (ii), state which solution contains the iron(II) ions.

Fe^{2+} ions are contained in solution

Explain how your observations support your conclusion.

.....
.....

- (iv) Aqueous EDTA is a reagent used to identify some transition metals. To 1 cm depth of the solution containing the nickel(II) ion, add 1 cm depth of aqueous EDTA.

observation

.....
.....

- (v) State what you would expect to **see** if acidified potassium manganate(VII) was added to a sample of the solution containing the iron(II) ion.
Do not carry out this experiment.

expected observation

.....

[6]

[Total: 15]

I	
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VI	

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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CHEMISTRY

9701/35

Advanced Practical Skills 1

May/June 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 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	
3	
Total	

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



You are advised to begin work on question 2, and return to question 1 later.

For
Examiner's
Use

- 1 In this experiment you are to investigate the reaction between phosphoric acid and sodium hydroxide in order to determine the chemical equation.

FA 1 is an aqueous solution of phosphoric acid, H_3PO_4 .

FA 2 is $0.115 \text{ mol dm}^{-3}$ sodium hydroxide, NaOH .

phenolphthalein indicator

(a) Method

Dilution

- Weigh the 100 cm^3 beaker provided. Record the mass in the space below.
- Use the measuring cylinder to add about 10 cm^3 of **FA 1** to the beaker. Weigh the beaker with **FA 1** and record the mass.
- Calculate the mass of **FA 1** used and record this in the space below.
- Pour the **FA 1** from the beaker into the 250 cm^3 graduated (volumetric) flask provided, labelled **FA 3**.
Wash the beaker twice with small quantities of water and add these washings to the volumetric flask.
- Make the solution up to the mark using distilled water. This diluted solution of phosphoric acid is **FA 3**.
- Ensure that the contents of the flask are thoroughly mixed before using **FA 3** for your titrations.

Titration

- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add 5 drops of phenolphthalein indicator to the flask. The indicator should remain colourless.
- Titrate **FA 3** with **FA 2** until the indicator changes to a permanent pale pink colour.
- 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 sure 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

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V	
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VII	

[7]

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

25.0 cm³ of **FA 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 hydroxide were present in the volume of **FA 2** calculated in (b).

moles of NaOH = ...mol

- (ii) The phosphoric acid, **FA 1**, that you weighed out contained 8.40 % by mass of H₃PO₄. Calculate the mass of H₃PO₄ that you weighed out.

mass of H₃PO₄ = ... g

- (iii) Calculate how many moles of H₃PO₄ were present in 25.0 cm³ of the diluted solution, **FA 3**.
(A_r : H, 1.0; O, 16.0; P, 31.0)

moles of H₃PO₄ = ... mol

- (iv) Use your answers to (i) and (iii) to calculate how many moles of NaOH react with 1 mole of H_3PO_4 .
Give your answer to the nearest whole number.

moles of NaOH = ... mol

- (v) When NaOH reacts with H_3PO_4 , the salt formed could be NaH_2PO_4 , Na_2HPO_4 or Na_3PO_4 .
Use your answer to (iv) to deduce which one of these three salts was the major product formed during the titration.
Write the equation for the reaction of NaOH with H_3PO_4 to produce this salt.

[5]

- (d) (i) A 25 cm^3 pipette is accurate to $\pm 0.06\text{ cm}^3$.
Calculate the maximum percentage error when the pipette was used to measure solution **FA 3**.

percentage error in measuring **FA 3** = ... %

- (ii) State the maximum error in the mass of the 100 cm^3 beaker used in the dilution of **FA 1**.

maximum error = ... g

- (iii) Calculate the maximum percentage error in the mass of **FA 1** used.

maximum percentage error = ... %
[2]

[Total: 15]

- 2 In this experiment you are to heat a hydrated salt, **FA 4**, to remove the water of crystallisation. You will then calculate the relative formula mass of the anhydrous salt. The formula of **FA 4** is $\text{MX}_2 \cdot 2\text{H}_2\text{O}$, where **M** is a metal and **X** is a halogen.

(a) Method

You will carry out the following experiment twice.

Record all weighings for the first and second experiments in an appropriate form in the space below.

- Record the mass of an empty crucible without its lid.
- Add 2.6 – 3.4 g of **FA 4** into the weighed crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently for about one minute with the lid off. Then heat strongly for a further **four** minutes.
- Put the lid on the crucible and leave to cool for several minutes.

While you are waiting for the crucible to cool, start work on Question 1.

- When the crucible is cool, **remove the lid** and weigh the crucible with the anhydrous residue, MX_2 .
- Repeat the experiment using the second crucible. This time add 1.6 – 2.4 g of **FA 4**.

I	
II	
III	
IV	
V	
VI	

[6]

(b) Calculation

Show your working and express your answers to **each** step to **three** significant figures.

- (i) Calculate the number of moles of water removed from the hydrated salt in the **first** experiment.
(A_r : H, 1.0; O, 16.0)

moles of H_2O = ... mol

- (ii) Complete the equation for the removal of water from hydrated **FA 4**.
Include state symbols.



- (iii) Use your equation from (ii) and your answer to (i) to calculate the number of moles of anhydrous MX_2 produced in the **first** experiment.

moles of $\text{MX}_2 = \text{..... mol}$

- (iv) Use your results from the **first** experiment to calculate the mass of anhydrous MX_2 produced.

mass of $\text{MX}_2 = \text{..... g}$

- (v) Calculate the relative formula mass of MX_2 .

relative formula mass of $\text{MX}_2 = \text{.....}$
[4]

- (c) (i) Suggest how the experimental procedure could be modified to ensure that all of the water of crystallisation had been removed by heating hydrated **FA 4**.

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

- (ii) The crucible was cooled with the lid on to prevent absorption of water vapour from the air. Suggest an alternative way of preventing water vapour being absorbed.

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

[Total: 12]

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.

No additional tests for ions present should be attempted.

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

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

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

(a) **FA 4** contains a halide ion.

FA 5 is a solution of a mixture of two salts. It contains **one cation** and **two anions**.

- Place a spatula measure of **FA 4** into a boiling tube.
- Half fill the boiling tube with distilled water and stir until the solid dissolves. Use this aqueous solution of **FA 4** as instructed in the following tests.

(i) Use aqueous silver nitrate and aqueous ammonia to identify the halide ion present in **FA 4**.

Carry out the same test on **FA 5**.

Use a 1 cm depth of solution in a test-tube for each test that you carry out.

Record your observations **and** conclusions in an appropriate form in the space below.

(ii) Write the **ionic** equation for the reaction of the halide ion in **FA 4** in test (i).

.....

- (iii) Use your conclusion from **3(a)(i)** and your answer to **2(b)(v)** to calculate the relative atomic mass of metal **M** in **FA 4**.
(A_r : F, 19.0; Cl, 35.5; Br, 79.9; I, 127)

(If you were unable to calculate the relative formula mass of anhydrous MX_2 in **2(b)(v)** assume that it was 222, so that you are able to carry out this calculation).

A_r of **M** = ...

- (iv) The relative atomic masses of some of the cations on page 10 are given below.
(A_r : Mg, 24.3; Ca, 40.0; Fe, 55.8; Cu, 63.5; Mn, 54.9; Zn, 65.4; Ba, 137; Pb, 207)

M is one of the cations listed above.

Suggest the identity of **M** and justify your answer.

.....
.....

- (v) Suggest why it was not necessary to include the cations aluminium and chromium from page 10 in the list of relative atomic masses in **(iv)**.

.....
.....

[7]

- (b) (i) Perform the experiments described below and record your observations in the table
Record your observations in the table below.

<i>test</i>	<i>observation</i>
To 1 cm depth of aqueous FA 4 in a test-tube, add aqueous ammonia.	
To 1 cm depth of FA 5 in a test-tube, add aqueous ammonia.	

- (ii) Use **only** the results from (i) to identify the **two** possible cations that would have resulted in your observations for **FA 4**.

FA 4 contains or

- (iii) Use the results from (i) to identify the cation in **FA 5**.

FA 5 contains.....

- (iv) Use the information on pages 10 and 11 to select **one** reagent, other than sodium hydroxide, to distinguish between the pair of possible cations in **FA 4** identified in (ii). Carry out the test with your selected reagent, using a 1 cm depth of **FA 4**. From **this** test, identify the cation in **FA 4**.

reagent

observation

FA 4 contains

- (v) Perform the tests described below and record your observations. Identify the second anion in **FA 5**.

<i>test</i>	<i>observation</i>
To 1 cm depth of aqueous FA 4 in a test-tube, add 1 cm depth of FA 5 .	
Then add 2 cm depth of dilute hydrochloric acid.	

FA 5 contains

[6]

[Total: 13]

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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