

CANDIDATE
NAME

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CENTRE
NUMBER

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CANDIDATE
NUMBER

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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

October/November 2014

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

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

Qualitative Analysis Notes are printed on pages 14 and 15.

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.

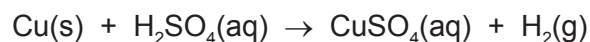
Session
Laboratory

For Examiner's Use	
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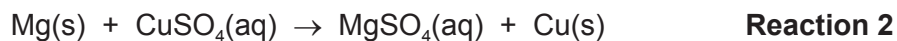
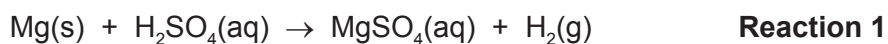
This document consists of **13** printed pages and **3** blank pages.



- 1 You are to determine the enthalpy change of reaction, ΔH , for the reaction shown below.



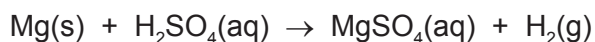
Since copper is an unreactive metal it does not react directly with dilute acids. You will therefore need to find the enthalpy change of reaction for two reactions that do occur. The equations for these two reactions are below.



You will carry out experiments to find the enthalpy changes for each of **Reaction 1** and **Reaction 2** and use these values to calculate the enthalpy change for the reaction of copper with sulfuric acid.

TURN OVER FOR EXPERIMENTAL METHOD

Determining the enthalpy change for Reaction 1



Reaction 1

(a) Method

FA 1 is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄.

FA 2 is magnesium powder, Mg.

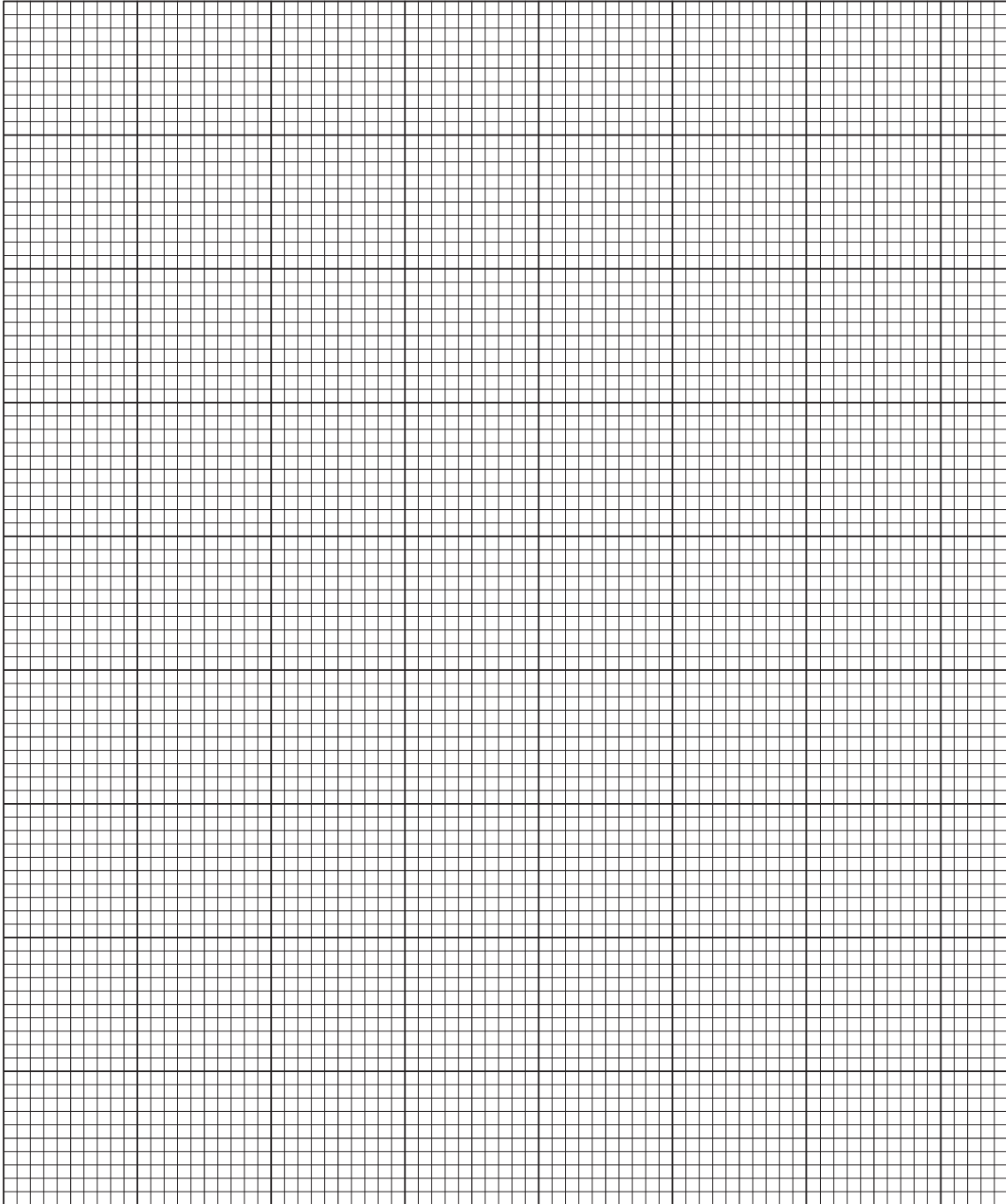
Read through the method before you start any practical work and prepare a suitable table for your results.

- Weigh the stoppered tube containing **FA 2**. Record the mass.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FA 1** into the plastic cup.
- Measure the temperature of **FA 1** in the plastic cup and start the stop clock. Record this temperature as being the temperature at time = 0.
- Measure, and record, the temperature of this **FA 1** every half minute for 2 minutes.
- At time = 2 $\frac{1}{2}$ minutes add the **FA 2** to the acid and stir carefully to reduce acid spray.
- Measure the temperature of the mixture in the cup at time = 3 minutes and then every half minute up to time = 7 minutes.
- Continue stirring occasionally throughout this time.
- Weigh the stoppered tube that had contained **FA 2**. Record the mass.
- Calculate and record the mass of **FA 2** added to the sulfuric acid.
- Rinse the plastic cup with water and shake to dry.

I	
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VI	

[6]

(b) (i) On the grid below plot a graph of temperature (y-axis) against time (x-axis).

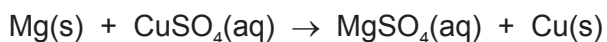


(ii) Complete the graph by inserting **two, straight** lines of best fit:

- one to show the temperature up to time = $2\frac{1}{2}$ minutes,
- one to show the temperature after time = $2\frac{1}{2}$ minutes.

I	
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III	
IV	

Determining the enthalpy change for Reaction 2



Reaction 2

(d) Method

FA 3 is 1.00 mol dm⁻³ copper(II) sulfate, CuSO₄.

FA 4 and **FA 5** are magnesium powder, Mg.

Read through the method before you start any practical work and prepare a suitable table for your results.

- Weigh the stoppered tube containing **FA 4**. Record the mass.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FA 3** into the plastic cup.
- Measure the temperature of **FA 3** in the plastic cup and record the temperature.
- Add the **FA 4** to the **FA 3** in the cup and stir the mixture constantly.
- Measure and record the maximum temperature reached during the reaction.
- Calculate and record the maximum temperature change that occurred during the reaction.
- Weigh the stoppered tube that had contained **FA 4**. Record the mass.
- Calculate and record the mass of **FA 4** added to the copper(II) sulfate.
- Empty the contents of the plastic cup into the 100 cm³ beaker labelled **waste**.
- Rinse the plastic cup and shake to dry.
- Repeat this experiment using **FA 5** in place of **FA 4**.

[2]

(e) Calculations

- (i) Using your results from (d), calculate the mean temperature rise.

mean temperature rise = ... °C

- (ii) Using your results from (d), calculate the mean mass of magnesium used.

mean mass = ... g

- (iii) Show, using a suitable calculation, that the copper(II) sulfate was in excess in these reactions.

- (iv) Using your values from (i) and (ii), calculate the enthalpy change, in kJ mol^{-1} , for the reaction between magnesium and copper(II) sulfate.

[Assume that 4.2 J is needed to raise the temperature of 1.0 cm^3 of solution by 1.0°C .]

[A_r : Mg, 24.3]



Reaction 2

enthalpy change for **Reaction 2** = ... kJ mol^{-1}
sign value

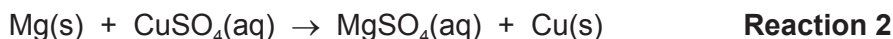
[4]

Enthalpy change for Reaction 3

Reaction 3 is shown below.



- (f) Use your values for the enthalpy changes for **Reactions 1** and **2** to calculate the enthalpy change for **Reaction 3**.



Show clearly how you obtained your answer.

(If you were unable to calculate the enthalpy changes for **Reactions 1** and **2**, you should assume that the value for **Reaction 1** is -444 kJ mol^{-1} and that the value for **Reaction 2** is -504 kJ mol^{-1} . Note: these are not the correct values.)

enthalpy change for **Reaction 3** = ... kJ mol^{-1}
sign *value*
 [2]

- (g) (i) The method you used to determine the enthalpy change for **Reaction 1** was more accurate than the method you used to determine the enthalpy change for **Reaction 2**. Suggest **two** reasons why the method used for **Reaction 2** was less accurate. Explain your answers.

1

2

- (ii) A student suggested that the accuracy of the method used for **Reaction 2** could be improved by using a larger volume of copper(II) sulfate. Is this a correct suggestion? Give a reason for your answer.

.....

 [3]

[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.

(a) **FA 6** is a solid that contains one cation and one anion. One of the ions present is included in the lists on pages 14 and 15. This ion contains the element nitrogen.

- (i) State which nitrogen-containing ions could be present. Select reagents for use in tests that would distinguish between them.

.....

.....

.....

- (ii) Carry out tests on **FA 6** using the reagents selected in (i) to identify the nitrogen-containing ion. Record your tests and observations in the space below.

(iii) Identify the nitrogen-containing ion in **FA 6**.

Ion present is

[5]

(b) Half fill the 250 cm³ beaker with water and heat the water to about 60 °C. This is the water bath to be used in one of the following tests.

Carry out the following tests on **FA 7** and complete the table below.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 7 in a test-tube, add a few drops of dilute hydrochloric acid.	
To a 1 cm depth of FA 7 in a test-tube, add a few drops of aqueous potassium iodide, then	
add aqueous ammonia.	
To a 1 cm depth of FA 7 in a test-tube, add a few drops of aqueous sodium hydroxide and then add aqueous ammonia dropwise, with gentle shaking, until the precipitate just dissolves, then	No observation required.
add one spatula measure of glucose and leave to stand in the hot water bath.	
When you have completed this test, dispose of the solution and rinse the test-tube.	

[4]

- (c) Solid **FA 8** contains one cation and one anion from those included in the lists on pages 14 and 15.

Carry out the following tests on **FA 8**. For each test record your observations.

- (i) In a hard-glass test-tube heat approximately half of the **FA 8**, gently at first and then more strongly. Leave to cool.

.....

.....

.....

- (ii) To a 2 cm depth of dilute nitric acid in a boiling tube, add the remaining **FA 8**.

Keep the solution obtained for tests (iii) and (iv).

.....

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- (iii) To a 1 cm depth of the solution from (ii) in a test-tube, add aqueous sodium hydroxide until no further change occurs.

.....

.....

.....

- (iv) To a 1 cm depth of the solution from (ii) in a test-tube, add aqueous ammonia until no further change occurs.

.....

.....

.....

- (v) Use your observations from (i) to (iv) to identify the ions present in **FA 8**.

Ions present and

[6]

[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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
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 $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colourless

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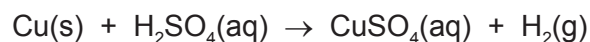
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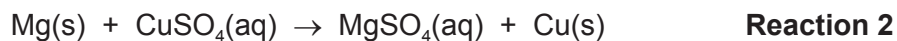
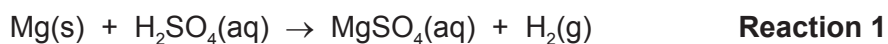
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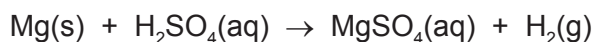
Since copper is an unreactive metal it does not react directly with dilute acids. You will therefore need to find the enthalpy change of reaction for two reactions that do occur. The equations for these two reactions are below.



You will carry out experiments to find the enthalpy changes for each of **Reaction 1** and **Reaction 2** and use these values to calculate the enthalpy change for the reaction of copper with sulfuric acid.

TURN OVER FOR EXPERIMENTAL METHOD

Determining the enthalpy change for Reaction 1



Reaction 1

(a) Method

FA 1 is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄.

FA 2 is magnesium powder, Mg.

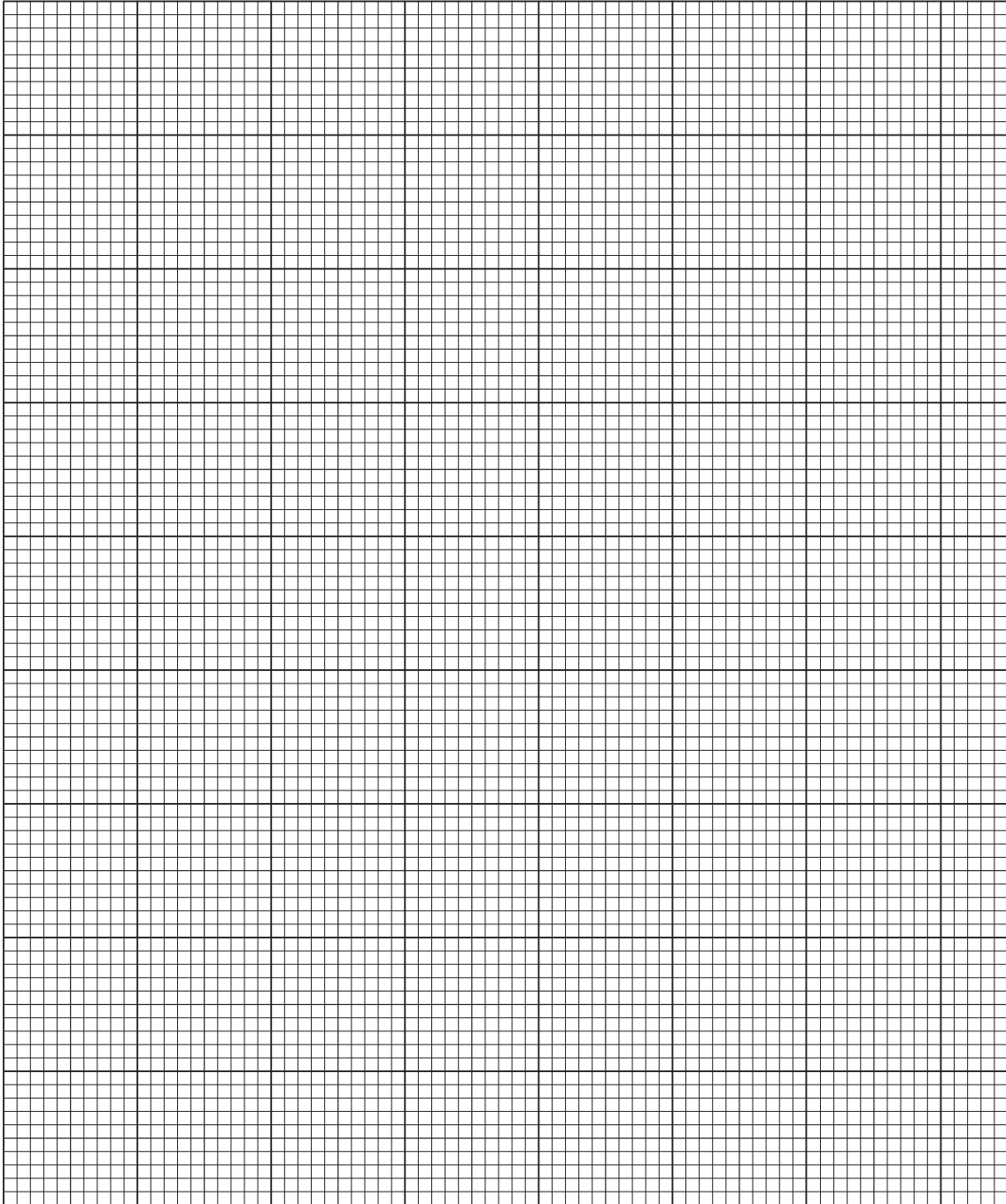
Read through the method before you start any practical work and prepare a suitable table for your results.

- Weigh the stoppered tube containing **FA 2**. Record the mass.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FA 1** into the plastic cup.
- Measure the temperature of **FA 1** in the plastic cup and start the stop clock. Record this temperature as being the temperature at time = 0.
- Measure, and record, the temperature of this **FA 1** every half minute for 2 minutes.
- At time = 2 $\frac{1}{2}$ minutes add the **FA 2** to the acid and stir carefully to reduce acid spray.
- Measure the temperature of the mixture in the cup at time = 3 minutes and then every half minute up to time = 7 minutes.
- Continue stirring occasionally throughout this time.
- Weigh the stoppered tube that had contained **FA 2**. Record the mass.
- Calculate and record the mass of **FA 2** added to the sulfuric acid.
- Rinse the plastic cup with water and shake to dry.

I	
II	
III	
IV	
V	
VI	

[6]

(b) (i) On the grid below plot a graph of temperature (y-axis) against time (x-axis).



(ii) Complete the graph by inserting **two, straight** lines of best fit:

- one to show the temperature up to time = $2\frac{1}{2}$ minutes,
- one to show the temperature after time = $2\frac{1}{2}$ minutes.

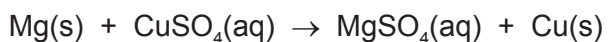
I	
II	
III	
IV	

(c) Calculations

- energy change = ... J

- $$\text{Mg(s)} + \text{H}_2\text{SO}_4\text{(aq)} \rightarrow \text{MgSO}_4\text{(aq)} + \text{H}_2\text{(g)} \quad \text{Reaction 1}$$

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Determining the enthalpy change for Reaction 2**Reaction 2****(d) Method**

FA 3 is 1.00 mol dm⁻³ copper(II) sulfate, CuSO₄.

FA 4 and **FA 5** are magnesium powder, Mg.

Read through the method before you start any practical work and prepare a suitable table for your results.

- Weigh the stoppered tube containing **FA 4**. Record the mass.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FA 3** into the plastic cup.
- Measure the temperature of **FA 3** in the plastic cup and record the temperature.
- Add the **FA 4** to the **FA 3** in the cup and stir the mixture constantly.
- Measure and record the maximum temperature reached during the reaction.
- Calculate and record the maximum temperature change that occurred during the reaction.
- Weigh the stoppered tube that had contained **FA 4**. Record the mass.
- Calculate and record the mass of **FA 4** added to the copper(II) sulfate.
- Empty the contents of the plastic cup into the 100 cm³ beaker labelled **waste**.
- Rinse the plastic cup and shake to dry.
- Repeat this experiment using **FA 5** in place of **FA 4**.

[2]

(e) Calculations

- (i) Using your results from (d), calculate the mean temperature rise.

mean temperature rise = ... °C

- (ii) Using your results from (d), calculate the mean mass of magnesium used.

mean mass = ... g

- (iii) Show, using a suitable calculation, that the copper(II) sulfate was in excess in these reactions.

- (iv) Using your values from (i) and (ii), calculate the enthalpy change, in kJ mol^{-1} , for the reaction between magnesium and copper(II) sulfate.

[Assume that 4.2 J is needed to raise the temperature of 1.0 cm^3 of solution by 1.0°C .]

[A_r : Mg, 24.3]



Reaction 2

enthalpy change for **Reaction 2** = ... kJ mol^{-1}
sign value

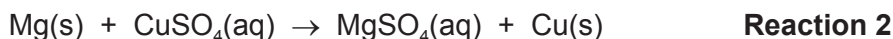
[4]

Enthalpy change for Reaction 3

Reaction 3 is shown below.



- (f) Use your values for the enthalpy changes for **Reactions 1** and **2** to calculate the enthalpy change for **Reaction 3**.



Show clearly how you obtained your answer.

(If you were unable to calculate the enthalpy changes for **Reactions 1** and **2**, you should assume that the value for **Reaction 1** is -444 kJ mol^{-1} and that the value for **Reaction 2** is -504 kJ mol^{-1} . Note: these are not the correct values.)

enthalpy change for **Reaction 3** = ... kJ mol^{-1}
sign *value* [2]

- (g) (i) The method you used to determine the enthalpy change for **Reaction 1** was more accurate than the method you used to determine the enthalpy change for **Reaction 2**. Suggest **two** reasons why the method used for **Reaction 2** was less accurate. Explain your answers.

1

2

- (ii) A student suggested that the accuracy of the method used for **Reaction 2** could be improved by using a larger volume of copper(II) sulfate. Is this a correct suggestion? Give a reason for your answer.

.....
 [3]

[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.

(a) **FA 6** is a solid that contains one cation and one anion. One of the ions present is included in the lists on pages 14 and 15. This ion contains the element nitrogen.

- (i) State which nitrogen-containing ions could be present. Select reagents for use in tests that would distinguish between them.

.....

.....

.....

- (ii) Carry out tests on **FA 6** using the reagents selected in (i) to identify the nitrogen-containing ion. Record your tests and observations in the space below.

(iii) Identify the nitrogen-containing ion in **FA 6**.

Ion present is

[5]

(b) Half fill the 250 cm³ beaker with water and heat the water to about 60 °C. This is the water bath to be used in one of the following tests.

Carry out the following tests on **FA 7** and complete the table below.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 7 in a test-tube, add a few drops of dilute hydrochloric acid.	
To a 1 cm depth of FA 7 in a test-tube, add a few drops of aqueous potassium iodide, then	
add aqueous ammonia.	
To a 1 cm depth of FA 7 in a test-tube, add a few drops of aqueous sodium hydroxide and then add aqueous ammonia dropwise, with gentle shaking, until the precipitate just dissolves, then	No observation required.
add one spatula measure of glucose and leave to stand in the hot water bath.	
When you have completed this test, dispose of the solution and rinse the test-tube.	

[4]

- (c) Solid **FA 8** contains one cation and one anion from those included in the lists on pages 14 and 15.

Carry out the following tests on **FA 8**. For each test record your observations.

- (i) In a hard-glass test-tube heat approximately half of the **FA 8**, gently at first and then more strongly. Leave to cool.

.....

.....

.....

- (ii) To a 2 cm depth of dilute nitric acid in a boiling tube, add the remaining **FA 8**.

Keep the solution obtained for tests (iii) and (iv).

.....

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- (iii) To a 1 cm depth of the solution from (ii) in a test-tube, add aqueous sodium hydroxide until no further change occurs.

.....

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.....

- (iv) To a 1 cm depth of the solution from (ii) in a test-tube, add aqueous ammonia until no further change occurs.

.....

.....

.....

- (v) Use your observations from (i) to (iv) to identify the ions present in **FA 8**.

Ions present and

[6]

[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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
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 $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colourless

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CHEMISTRY

9701/34

Paper 3 Advanced Practical Skills 2

October/November 2014

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

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

Qualitative Analysis Notes are printed on pages 10 and 11.

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

Session	
Laboratory	

For Examiner's Use	
1	
2	
3	
Total	

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



- 1 Hydrogen peroxide, H_2O_2 , is used in hair bleach and for skin therapies. In this experiment you will determine the concentration of a solution of hydrogen peroxide by titration with acidified potassium manganate(VII).

FB 1 is $0.0250 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FB 2 is dilute sulfuric acid, H_2SO_4 .

FB 3 is aqueous hydrogen peroxide, H_2O_2 .

(a) Method

Dilution of FB 3

- Pipette **25.0 cm³** of **FB 3** into the volumetric (graduated) flask.
- Make the solution up to the mark using distilled water.
- Shake the flask thoroughly.
- This diluted solution of hydrogen peroxide is **FB 4**.

Titration

- Fill the burette with **FB 1**.
- Pipette **10.0 cm³** of **FB 4** into a conical flask.
- Use a measuring cylinder to add 25 cm^3 of **FB 2** into the same flask.
- Add **FB 1** until a permanent pale pink colour is seen.
- 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 **FB 1** added in each accurate titration.

Keep solution **FB 2** for use in Question 3 and solution **FB 3** for use in Questions 2 and 3.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

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

10.0 cm³ of **FB 4** required cm³ of **FB 1**. [1]

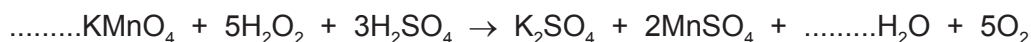
(c) Calculations

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

- (i) Calculate the number of moles of potassium manganate(VII) present in the volume calculated in (b).

moles of KMnO₄ = ... mol

- (ii) Complete the equation below for the reaction of potassium manganate(VII) with hydrogen peroxide. State symbols are not required.



- (iii) Use your answers to (i) and (ii) to calculate the number of moles of hydrogen peroxide used in each titration.

moles of H₂O₂ = ... mol

- (iv) Calculate the concentration of H₂O₂ in **FB 4**, in mol dm⁻³.

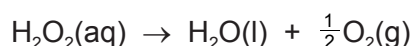
concentration of H₂O₂ in **FB 4** = ... mol dm⁻³

- (v) Calculate the concentration of H₂O₂ in **FB 3**, in mol dm⁻³.

concentration of H₂O₂ in **FB 3** = ... mol dm⁻³
[5]

[Total: 13]

- 2 In this experiment you will determine the enthalpy change, ΔH , for the catalytic decomposition of hydrogen peroxide into water and oxygen.



FB 3 is aqueous hydrogen peroxide, H_2O_2 .

FB 5 is manganese(IV) oxide, MnO_2 , the catalyst for the decomposition.

(a) Method

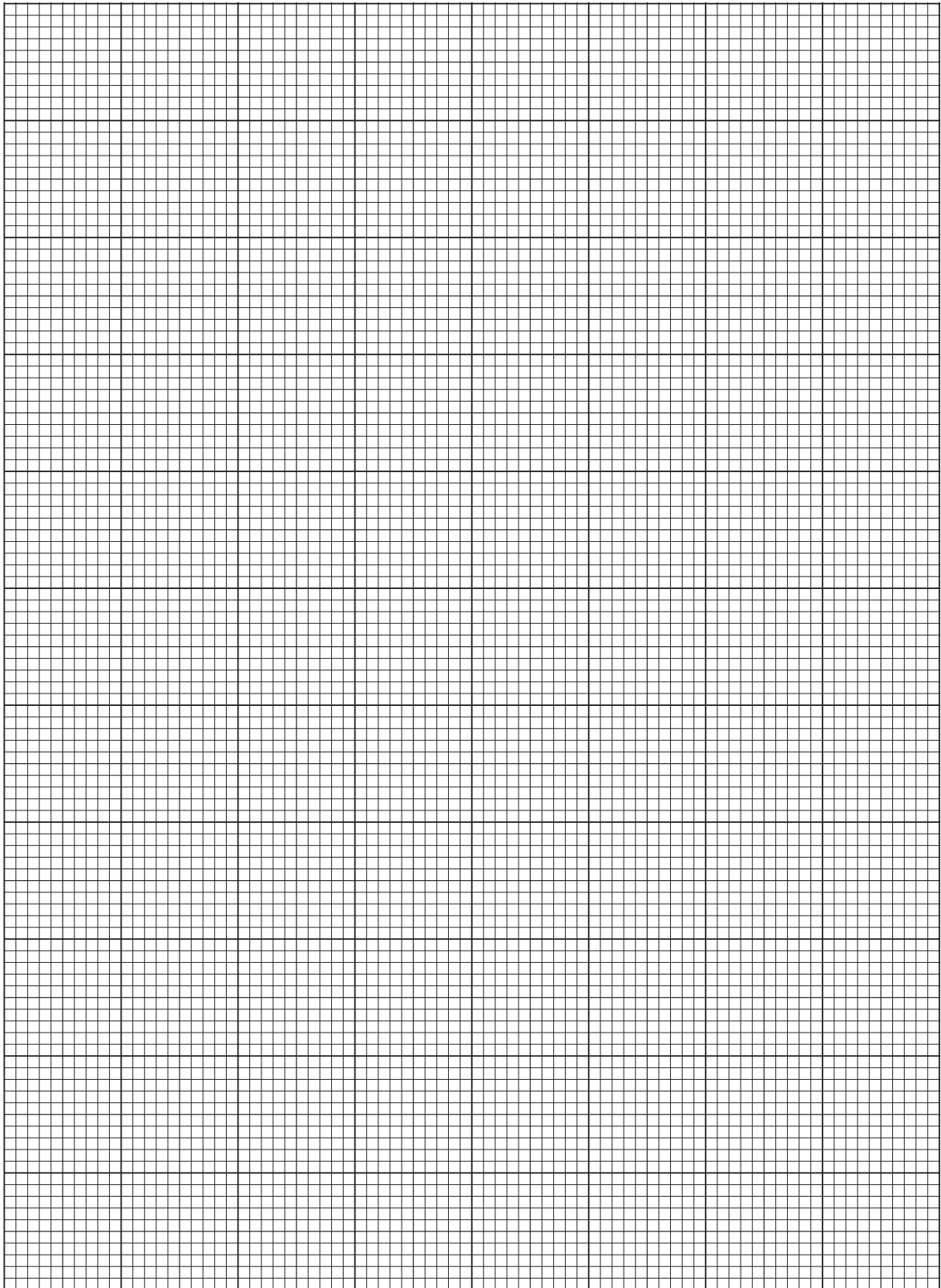
Read through the method before starting any practical work and prepare a table for your results in the space below.

- For the **first** experiment, support the plastic cup inside the 250 cm³ beaker.
 - Use a measuring cylinder to transfer 40 cm³ of distilled water into the plastic cup.
 - Use a measuring cylinder to add 10 cm³ of **FB 3** into the plastic cup.
 - Measure and record the initial temperature of the mixture.
 - Add a heaped spatula measure of **FB 5** to the mixture in the plastic cup.
 - Stir constantly until the maximum temperature is reached and record this temperature.
 - Calculate and record the temperature rise.
 - Wash and wipe out your plastic cup and rinse the thermometer, ready for the second experiment.
-
- For the **second** experiment, support the plastic cup inside the 250 cm³ beaker.
 - Use a measuring cylinder to transfer 30 cm³ of distilled water into the plastic cup.
 - Use a measuring cylinder to add 20 cm³ of **FB 3** into the plastic cup.
 - Measure and record the initial temperature of the mixture.
 - Add a heaped spatula measure of **FB 5** to the mixture in the plastic cup.
 - Stir constantly until the maximum temperature is reached and record this temperature.
 - Calculate and record the temperature rise.
 - Wash and wipe out your plastic cup and rinse the thermometer, ready for the third experiment.
-
- Carry out the **third** experiment in a similar way.
 - Transfer 20 cm³ of distilled water into the plastic cup.
 - Add 30 cm³ of **FB 3** into the plastic cup.
 - Measure and record the initial temperature of the mixture.
 - Add a heaped spatula measure of **FB 5** to the mixture in the plastic cup.
 - Record the maximum temperature, then calculate and record the temperature rise.
-
- For the **fourth** experiment, use 10 cm³ of distilled water and 40 cm³ of **FB 3**.

I	
II	
III	
IV	
V	

[5]

- (b) Using the grid below, plot a graph of the temperature rise (y-axis) against the volume of **FB 3** (x-axis).
Draw the line of best fit.



I	
II	
III	
IV	

[4]

(c) Calculation

- (i) Use your graph to calculate the average temperature rise for each 1.0 cm³ of **FB 3** used. Show your working clearly on the graph.

average temperature rise = ... °C

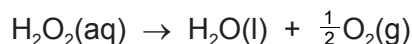
- (ii) Calculate the energy released for each 1.0 cm³ of **FB 3** used.
(Assume that 4.2 J are needed to raise the temperature of 1.0 cm³ of solution by 1.0 °C.)

energy released = ... J

- (iii) Use your answer to **1(c)(v)** to calculate the number of moles of hydrogen peroxide in 1.0 cm³ of **FB 3**.
(If you were unable to calculate the concentration of H₂O₂ in **FB 3**, assume that it was 1.72 mol dm⁻³. Note: this is not the correct value.)

number of moles of H₂O₂ = ... mol

- (iv) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction below.



enthalpy change = ... kJ mol⁻¹
(sign) (value) [4]

- (d) Which **one** of the four experiments that you carried out is likely to be the least accurate? Explain your choice.

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

[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.

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 will carry out further experiments with aqueous hydrogen peroxide, **FB 3**.

FB 2 is dilute sulfuric acid, H_2SO_4 .

FB 3 is aqueous hydrogen peroxide, H_2O_2 .

FB 6 is a solution containing two cations and one anion.

FB 7 is a solution containing one cation and one anion.

- (i) To a 1 cm depth of **FB 6** in a boiling tube, add aqueous sodium hydroxide until it is in excess. Then heat the tube, gently and with care. Keep the mixture for test (ii). Record your observations. Identify the cations in **FB 6**.

observations

.....

.....

FB 6: cations are and

- (ii) To the mixture obtained from (i) add a 1 cm depth of **FB 3**. Shake the tube. Record your observations.

observations

.....

.....

What **type** of reaction has taken place? Explain your answer.

.....

.....

- (iii) To a 1 cm depth of **FB 3** in a test-tube, add an equal volume of sulfuric acid, **FB 2**. Then add a 1 cm depth of **FB 7**, followed by a few drops of starch solution. Record all your observations. Draw what conclusions you can about the ions in **FB 7**. If no conclusion is possible, write 'not known'.

observations

.....

.....

FB 7: cation anion [7]

- (b) **FB 7**, **FB 8** and **FB 9** are aqueous solutions, each containing one cation and one anion. Note that **FB 7** was also used in (a)(iii).

- (i) Carry out the following tests in test-tubes. Use 1 cm depths of solutions. Complete the table by recording your observations.

test	observations		
	FB 7	FB 8	FB 9
add a 2 cm strip of magnesium ribbon			
FB 7			
FB 8			

- (ii) Suggest the identify of the cation in **FB 9**.
Explain your answer.

cation

explanation

- (iii) Give the ionic equation for the reaction between **FB 7** and **FB 9**.

.....

- (iv) From your observations, identify **FB 8**.

.....

[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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
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 $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colourless

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CHEMISTRY

9701/35

Paper 3 Advanced Practical Skills 1

October/November 2014

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

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

Qualitative Analysis Notes are printed on pages 12 and 13.

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

Session
Laboratory

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1	
2	
3	
Total	

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

- 1 In this experiment you are to determine **x** in the formula of hydrated sodium carbonate, $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$, by titration.

FA 1 is aqueous sodium carbonate, Na_2CO_3 , containing 150.0 g of solid $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$ in each dm^3 of solution.

FA 2 is $0.110 \text{ mol dm}^{-3}$ nitric acid, HNO_3 .
methyl orange indicator

(a) Method

Diluting the solution of FA 1

- Pipette 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric (graduated) flask.
- Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of sodium carbonate is **FA 3**.
- Rinse the pipette with distilled water.

Titration

- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Fill the burette with **FA 2**.
- Add several drops of methyl orange indicator to the conical flask.
- Carry out 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.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

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

volume of **FA 2** = ... cm³
[1]

(c) Calculations

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

- (i) Calculate the number of moles of nitric acid in the volume of **FA 2** calculated in (b).

moles of HNO₃ = ... mol

- (ii) Complete the equation below and include the missing state symbols.



- (iii) Calculate the number of moles of sodium carbonate in 25.0 cm³ of solution **FA 3**.

moles of Na₂CO₃ in 25.0 cm³ of **FA 3** = ... mol

- (iv) Calculate the number of moles of sodium carbonate in 250.0 cm³ of solution **FA 3**.

moles of Na₂CO₃ in 250.0 cm³ of **FA 3** = ... mol

- (v) Use the information on page 2 and your answer to (iv) to calculate the relative formula mass, M_r , of hydrated sodium carbonate, $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$.

M_r of $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$ = ...

- (vi) Calculate the value of x in $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$. Give your answer to the nearest whole number.
[A_r : H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

x = ...
[6]

- (d) (i) State the maximum error in any single reading of the burette.

maximum error = ... cm^3

- (ii) Calculate the maximum percentage error in the volume of **FA 2** in your **first** accurate titre.

maximum percentage error = ...%
[1]

[Total: 15]

- 2 In this experiment you will determine the enthalpy change, ΔH , for the dehydration of hydrated sodium carbonate to anhydrous sodium carbonate.



In order to do this, first you will determine the enthalpy changes for the reactions of anhydrous sodium carbonate and hydrated sodium carbonate with excess hydrochloric acid. Then you will use Hess' Law to calculate the enthalpy change for the reaction above.

FA 4 is anhydrous sodium carbonate, Na_2CO_3 .

FA 5 is hydrated sodium carbonate, $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$.

FA 6 is 4.0 mol dm^{-3} hydrochloric acid, HCl .

- (a) Determination of the enthalpy change for the reaction of anhydrous sodium carbonate, **FA 4**, with excess hydrochloric acid.

Method

- Support a plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 25 cm^3 of **FA 6** into the plastic cup.
- Record the initial temperature of the acid.
- Weigh the container with **FA 4** and record the mass.
- **Slowly** add all the **FA 4** to the acid in the plastic cup.
Note that the reaction will be vigorous. Avoid inhaling any acid spray produced.
- Stir constantly until the maximum temperature is reached.
Record the maximum temperature.
- Weigh the container from which you added **FA 4** and record the mass.
- Calculate and record the mass of **FA 4** used and the temperature rise.

(b) Calculation

- (i) Calculate the energy produced during this reaction.
[Assume that 4.2 J are needed to raise the temperature of 1.0 cm³ of solution by 1.0 °C.]

energy produced = ... J

- (ii) Calculate the number of moles of anhydrous sodium carbonate used.
[A_r: C, 12.0; O, 16.0; Na, 23.0]

moles of Na_2CO_3 = ... mol

- (iii) Calculate the enthalpy change, in kJ mol^{-1} , for the reaction below.



enthalpy change for anhydrous Na_2CO_3 = ... kJ mol⁻¹

sign value

[3]

- (c) Determination of the enthalpy change for the reaction of hydrated sodium carbonate, **FA 5**, with excess hydrochloric acid.

Method

- Support the second plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FA 6** into the plastic cup.
- Record the initial temperature of the acid.
- Weigh the container with **FA 5** and record the mass.
- Slowly add all the **FA 5** to the acid in the plastic cup.
Note that the reaction will be vigorous. Avoid inhaling any acid spray produced.
- Stir constantly until the minimum temperature is reached.
Record the minimum temperature.
- Weigh the container from which you added **FA 5** and record the mass.
- Calculate and record the mass of **FA 5** used and the temperature decrease.

[2]

(d) Calculation

- (i) Calculate the energy absorbed during this reaction.
[Assume that 4.2 J are absorbed when the temperature of 1.0 cm³ of solution falls by 1.0 °C.]

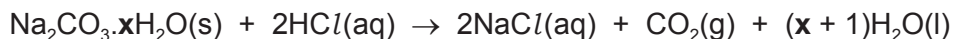
energy absorbed = ... J

- (ii) Use your answer to **1(c)(vi)** to calculate the number of moles of hydrated sodium carbonate, Na₂CO₃·xH₂O, used.
[A_r: H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

(If you were unable to calculate **x** in **1(c)(vi)**, you may assume that **x** = 8. Note: this is not the correct value.)

moles of Na₂CO₃·xH₂O = ... mol

- (iii) Calculate the enthalpy change, in kJ mol^{-1} , for the reaction below.



enthalpy change for $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O} = \dots\dots\dots \text{kJ mol}^{-1}$
sign *value*

[2]

- (e) Use your values for the enthalpy changes calculated in (b)(iii) and (d)(iii) to calculate the enthalpy change for the reaction below.



Show clearly how you obtained your answer.

(If you were unable to calculate the enthalpy changes, you should assume that the value in (b)(iii) is $-43.1 \text{ kJ mol}^{-1}$ and the value in (d)(iii) is $+32.8 \text{ kJ mol}^{-1}$. Note: these are not the correct values.)

enthalpy change = $\dots\dots\dots \text{kJ mol}^{-1}$
sign *value*

[2]

- (f) A student suggested that the experiments carried out in (a) and (c) would be more accurate if double the volume of hydrochloric acid had been used, but with hydrochloric acid of concentration 2.0 mol dm^{-3} , instead of 4.0 mol dm^{-3} .

State and explain whether or not you agree with the student's suggestion.

.....

 [1]

[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) **FA 7** and **FA 8** are mixtures. Each mixture contains one cation and two anions from those listed on pages 12 and 13.

Carry out the following tests and record all your observations in the table below.

<i>test</i>	<i>observations</i>	
	FA 7	FA 8
(i) Place a spatula measure of FA 7 or FA 8 into a boiling tube, then add approximately 5 cm³ of dilute nitric acid using the measuring cylinder. (Do not use FA 2 as the dilute nitric acid.) Then		
add about 5 cm³ of distilled water and shake to mix the solution. Use a 1 cm depth of the solution obtained in a test-tube for each of tests (ii) – (v).	No observation required.	
(ii) Add aqueous sodium hydroxide.		Do not carry out this test.
(iii) Add aqueous ammonia.		Do not carry out this test.
(iv) Add aqueous barium nitrate followed by dilute nitric acid.		
(v) Add aqueous silver nitrate followed by aqueous ammonia.		

- (vi) Use your observations to identify the following ions.

FA 7 contains cation

FA 7 contains anions and

FA 8 contains anions and

State and explain **all** the evidence for your identification of the cation in **FA 7**.

.....

.....

.....

[7]

- (b) **FA 9** contains two cations from the list on page 12.

- (i) Transfer approximately half of the **FA 9** into a hard-glass test-tube and heat gently at first, and then strongly, until no further change is seen.
Test with litmus papers while you are heating.
Record all your observations below.

.....

.....

.....

.....

.....

- (ii) Carry out further tests that will enable you to identify **both** cations in **FA 9**.
Describe your tests briefly and state your observations.

FA 9 contains cations and

[5]

[Total: 12]

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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
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 $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colourless

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CHEMISTRY

9701/36

Paper 3 Advanced Practical Skills 2

October/November 2014

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

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

Qualitative Analysis Notes are printed on pages 10 and 11.

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

Session	
Laboratory	

For Examiner's Use	
1	
2	
3	
Total	

This document consists of **10** printed pages and **2** blank pages.

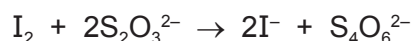


- 1 You are to determine the concentration of a solution of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

To do this you will first produce a known amount of iodine by reacting iodate(V) ions, IO_3^- , with an excess of iodide ions, I^- . The equation for this reaction is below.



The amount of iodine produced in this reaction can be found by titrating with thiosulfate ions. The equation for this reaction is below.



FB 1 is aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FB 2 is aqueous potassium iodate(V) containing 3.60 g dm^{-3} KIO_3 .

FB 3 is sulfuric acid, H_2SO_4 .

FB 4 is aqueous potassium iodide, KI .

starch indicator

(a) Method

- Fill a burette with **FB 1**.
- Pipette 25.0 cm^3 of **FB 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FB 3** into the conical flask.
- Use the measuring cylinder to add 10 cm^3 of **FB 4** into the conical flask. Brown iodine solution is produced.
- Add **FB 1** from the burette until most of the iodine has been removed and the solution in the conical flask is yellow.
- Add 10 drops of starch indicator to the contents of the conical flask. The solution will turn blue-black.
- Continue adding **FB 1**, from the burette, until the blue-black colour just disappears.
- Carry out 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.

I	
II	
III	
IV	
V	
VI	

[6]

- (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 **FB 2** required cm³ of **FB 1**. [1]

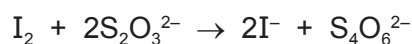
(c) Calculations

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

- (i) Calculate the number of moles of KIO₃ present in 25.0 cm³ of **FB 2**.
[A_r: O, 16.0; K, 39.1; I, 126.9]

moles of KIO₃ = ... mol

- (ii) The equations for the production of iodine and its titration with thiosulfate are shown below.



Use these equations to calculate the number of moles of thiosulfate present in the volume of **FB 1** you calculated in (b).

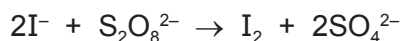
moles of S₂O₃²⁻ = ... mol

- (iii) Calculate the concentration, in mol dm⁻³, of sodium thiosulfate in **FB 1**.

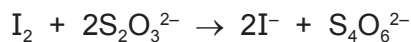
concentration = ... mol dm⁻³
[5]

[Total: 12]

- 2 In Question 1 iodide ions were oxidised by iodate(V) ions. Iodide ions can also be oxidised by peroxodisulfate ions, $\text{S}_2\text{O}_8^{2-}$, and you are to investigate the rate of this reaction.



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



After all the thiosulfate has reacted, the iodine turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the mixture to turn blue-black.

The rate of the reaction can be represented by rate = $\frac{1}{\text{reaction time}}$.

(a) Method

FB 4 is aqueous potassium iodide, KI.

FB 5 is $0.020 \text{ mol dm}^{-3}$ potassium peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$.

FB 6 is $0.010 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

Read through the method before you start any practical work and prepare a suitable table for your results in the space at the top of page 5.

- Empty and wash thoroughly the burette that you used in Question 1.

Experiment 1

- Use the measuring cylinder to add 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Use the 10 cm^3 pipette to add 10.0 cm^3 of **FB 6** to the beaker.
- Add 10 drops of starch indicator to the beaker.
- Fill a burette with **FB 5**.
- Run 20.00 cm^3 of **FB 5** 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 goes blue-black.
- Record this reaction time to the nearest second.
- Wash out both beakers and shake dry.

Experiment 2

- Use the measuring cylinder to add 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Use the 10 cm^3 pipette to add 10.0 cm^3 of **FB 6** to the beaker.
- Add 10 drops of starch indicator to the beaker.
- Fill the second burette with distilled water.
- Run 10.00 cm^3 of **FB 5** into the second 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into this second beaker containing **FB 5**.
- 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 goes blue-black.
- Record this reaction time to the nearest second.
- Wash out both beakers and shake dry.

Record your results in the space below. You should show the volume of **FB 5**, the volume of water and the reaction time for each experiment.

[3]

- (b) Carry out **three** further experiments to investigate how the reaction time changes with different volumes of peroxodisulfate. Remember that the combined volume of peroxodisulfate solution, **FB 5**, and distilled water must always be 20.00 cm³. Do not use a volume of **FB 5** that is less than 4.00 cm³.

Record the volume of **FB 5**, the volume of water and the reaction time for each experiment.

I	
II	
III	
IV	

[4]

- (c) Use your results from (a) and (b) to complete the table below. You should show the volume of **FB 5**, the reaction time and the volume of **FB 5** × reaction time.

[2]

- (d) A student who had carried out these experiments concluded that the rate of reaction was directly proportional to the volume of **FB 5**.

Using your values from (c), explain whether your results agree with this conclusion.

.....

.....

.....

..... [1]

- (e) Another student thought that the experiment could be made more accurate by giving longer reaction times. To do this he repeated **Experiment 1** with the same volumes but using $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate instead of **FB 6**. He found that the reaction never turned blue-black. Explain why.

.....

.....

..... [1]

- (f) Describe how you could modify this experiment to investigate the effect of the concentration of iodide ions, **FB 4**, on the rate of the reaction.

.....

.....

.....

..... [2]

[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.

Half fill the 250 cm³ beaker with water and heat it until the water is approximately 60 °C. Leave to stand. This water will be used as a water bath. Turn off the Bunsen burner when the water is hot.

(a) FB 7 and FB 8 are aqueous solutions. One of the elements present in the different ions in FB 7 and FB 8 is the same.

Carry out the following tests on **FB 7** and **FB 8** and complete the table.

<i>test</i>	<i>observations</i>
(i) To a 1cm depth of FB 7 in a test-tube add a 1cm depth of aqueous silver nitrate.	
(ii) To a 1cm depth of FB 7 in a test-tube add aqueous sodium hydroxide.	
(iii) To a 1cm depth of FB 7 in a test-tube add a 1cm depth of aqueous sodium hydroxide and then a 1cm depth of hydrogen peroxide. Leave to stand.	
(iv) Before starting this test, the Bunsen burner must be turned off. To a 2cm depth of dilute sulfuric acid add a few drops of FB 8 then add a 1 cm depth of ethanol. Leave to stand in the water bath.	

[5]

(b) What element is present in both **FB 7** and **FB 8**?

..... [1]

(c) Solid **FB 9** and aqueous solution **FB 10** both contain the same cation.

(i) Transfer approximately half of the **FB 9** into a hard-glass test-tube and heat.

observations

.....

(ii) Transfer the remaining **FB 9** into a boiling tube and carefully add dilute sulfuric acid.

observations

.....

(iii) To a 1 cm depth of **FB 10** in a test-tube, add a piece of magnesium ribbon. Leave to stand.

observations

.....

(iv) To a 1 cm depth of **FB 10** in a test-tube, add an equal depth of concentrated hydrochloric acid (**care: this is very corrosive**).

observations

.....

(v) Suggest which cation is present in both **FB 9** and **FB 10**.

.....

(vi) What change in the oxidation state of magnesium is occurring during reaction (c)(iii)?

Oxidation state change from to

[8]

(d) In which part of the Periodic Table are the elements identified as being present in **FB 7**, **FB 8**, **FB 9** and **FB 10**?

..... [1]

[Total: 15]

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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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
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 $\text{Ba}^{2+}(\text{aq})$ (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 manganate(VII) from purple to colourless

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