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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

October/November 2016

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.
A copy of the Periodic Table is printed on page 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	
3	
Total	

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

- 1 In **Questions 1** and **2** you will determine the percentage purity of industrial grade calcium carbonate, CaCO_3 , by two different methods.

In the first method you will collect and measure the volume of gas given off in the reaction between a known mass of industrial grade calcium carbonate, in the form of small marble chips, and a known amount of dilute hydrochloric acid. The acid will be in excess. The impurities in the calcium carbonate will not react with the acid.



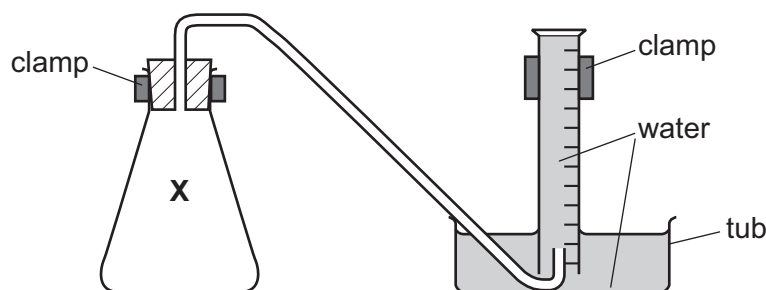
FA 1 is industrial grade calcium carbonate, CaCO_3 , in the form of small marble chips.

FA 2 is 2.00 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Read through the whole method before starting any practical work.

The diagram below may help you in setting up your apparatus.



- Fill the tub with water to a depth of about 5 cm.
- Fill the 250 cm^3 measuring cylinder **completely** with water. Hold a piece of paper towel firmly over the top, invert the measuring cylinder and place it in the water in the tub.
- Remove the paper towel and clamp the inverted measuring cylinder so the open end is in the water just above the base of the tub.
- Pipette 25.0 cm^3 of **FA 2** into the reaction flask labelled **X**.
- Check that the bung fits tightly in the neck of flask **X**, clamp flask **X** and place the end of the delivery tube into the inverted 250 cm^3 measuring cylinder.
- Weigh the container with **FA 1** and record the mass in the space on page 3.
- Remove the bung from the neck of the flask. Tip **FA 1** into the acid and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents. Swirl the flask occasionally until no more gas is evolved. Replace the flask in the clamp.
- Reweigh the container and any residue of **FA 1** and record the mass in the space on page 3.
- Calculate and record in the space on page 3 the mass of **FA 1** used.
- When no more gas is given off, measure and record the final volume of gas in the measuring cylinder in the space on page 3.

Keep the contents of flask X for use in Question 2.

Results

[2]

(b) 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 carbon dioxide gas collected in the measuring cylinder. (Assume that 1 mole of gas occupies 24.0 dm^3 under these conditions.)

moles of CO_2 = ... mol

- (ii) Use your answer to (i) and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO_3 = ... g

- (iii) Use your answer to (ii) and the mass of marble chips used in (a) to calculate a value for the percentage purity of the sample of industrial grade calcium carbonate, **FA 1**.

percentage purity of **FA 1** = ... %
[4]

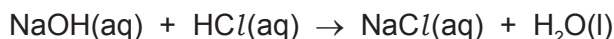
- (c) Not all the carbon dioxide given off in the reaction is collected in the measuring cylinder.

Suggest a change to the method which would lead to an increase in the volume of carbon dioxide collected.

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

[Total: 7]

- 2 You will determine the amount of hydrochloric acid remaining in flask **X** after the reaction with the marble chips in **Question 1**. You will do this by titration with sodium hydroxide of known concentration.



The impurities in the calcium carbonate will not react with the alkali.

FA 3 is 0.140 mol dm⁻³ sodium hydroxide, NaOH.
bromophenol blue indicator

(a) Method

- Transfer **all** the contents of flask **X** into the 250 cm³ volumetric flask.
- Rinse flask **X** with distilled water and add the washings to the volumetric flask. Add distilled water up to the mark.
- Stopper the volumetric flask and mix the contents thoroughly. Label this solution **FA 4**.
- Rinse the pipette then use it to transfer 25.0 cm³ of **FA 4** into a conical flask.
- Add about 10 drops of bromophenol blue indicator.
- Fill the burette with **FA 3**.
- 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.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 3** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b)** From your accurate titration results, obtain a suitable value for the volume of **FA 3** to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 4** required cm³ of **FA 3**. [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 sodium hydroxide, NaOH, present in the volume of **FA 3** you calculated in **(b)**.

moles of NaOH = ... mol

- (ii) Use your answer to (i) and the equation on page 4 to determine the number of moles of hydrochloric acid, HCl, present in the 25.0 cm³ of **FA 4** pipetted in **(a)**.

moles of HCl = ... mol

- (iii) Use your answer to (ii) to calculate the number of moles of hydrochloric acid, HCl, remaining in flask **X** after the reaction in **1(a)**.

moles of HCl remaining = ... mol

- (iv) Use the relevant information on page 2 to calculate the number of moles of hydrochloric acid, HCl, pipetted into flask **X** in **1(a)**.

moles of HCl pipetted into flask **X** = ... mol

- (v) Use your answers to (iii) and (iv) to calculate the number of moles of hydrochloric acid, HCl, which reacted with the marble chips in flask **X**.

moles of HCl which reacted in flask **X** = ... mol

- (vi) Use your answer to (v), the equation in **Question 1** and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate, CaCO_3 , in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO_3 = ... g

- (vii) Use your answer to (vi) and the mass of marble chips recorded in **1(a)** to calculate the percentage purity of **FA 1**.

percentage purity of **FA 1** = ... %
[5]

- (d) You have carried out two different methods to find the percentage purity of industrial grade calcium carbonate.

A source of error in **Question 1** is that some carbon dioxide escapes before the bung can be inserted.

How would this affect the percentage purity of **FA 1** calculated in the two questions? Explain your answers.

Question 1

.....

.....

.....

Question 2

.....

.....

.....

[3]

[Total: 16]

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) **FA 5** and **FA 6** are solids each containing one cation and one anion.
Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>	
	FA 5	FA 6
(i) Place a spatula measure of solid in a hard-glass test-tube and heat gently at first, then		
heat strongly until no further change takes place.		
Leave the tube to cool completely then add a 2 cm depth of dilute sulfuric acid to the solid residue. Shake the contents of the tube then leave it to stand.		

test	observations	
	FA 5	FA 6
(ii) Place a spatula measure of solid in a boiling tube and add a 2 cm depth of dilute sulfuric acid.		
Keep the solutions formed in (ii) for tests (iii) and (iv).		
(iii) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous sodium hydroxide.		
(iv) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous ammonia.		

- (v) Identify as many ions as you can from your observations. Write 'unknown' where you have not been able to identify an ion.

FA 5: cation anion

FA 6: cation anion

- (vi) Write an equation, including state symbols, for the reaction between **FA 6** and dilute sulfuric acid.

.....
[12]

(b) **FA 7** is a solution containing one anion from those listed on page 11. The anion is either a halide or contains nitrogen.

(i) You are to select suitable reagents to determine the identity of this anion. Record these in a suitable form below.

(ii) Use these reagents to carry out tests to identify the anion in **FA 7**.

Record your observations and conclusions in the space below.

[5]

[Total: 17]

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	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, $\text{SO}_3^{2-}(\text{aq})$	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

The Periodic Table of Elements

Group																									
1	2	Key												13	14	15	16	17	18						
		atomic number atomic symbol name relative atomic mass												1 H hydrogen 1.0											
3	4																								
Li lithium 6.9	Be beryllium 9.0																								
11	12																								
Na sodium 23.0	Mg magnesium 24.3																								
19	20																								
K potassium 39.1	Ca calcium 40.1																								
37	38																								
Rb rubidium 85.5	Sr strontium 87.6																								
55	56																								
Cs caesium 132.9	Ba barium 137.3																								
87	88																								
Fr francium —	Ra radium —																								

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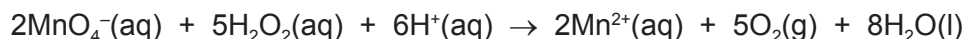
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Session
Laboratory

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 , can be oxidised to give oxygen, O_2 . This reaction happens rapidly in the presence of acidified potassium manganate(VII), KMnO_4 .



You will determine the concentration of a solution of hydrogen peroxide. You will first dilute the solution and then carry out a titration using acidified potassium manganate(VII), KMnO_4 .

FA 1 is aqueous hydrogen peroxide, H_2O_2 .

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

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

(a) Method

Dilution

- Pipette 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric flask.
- Add distilled water to make 250 cm^3 of solution and shake the flask thoroughly.
- Label this diluted solution of hydrogen peroxide **FA 4**.

Titration

- Fill the burette with **FA 2**.
- Rinse the pipette thoroughly with distilled water and then with a little **FA 4**.
- Pipette 25.0 cm^3 of **FA 4** into a conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 3** to the conical flask.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 2** added in each accurate titration.

Keep **FA 3** and **FA 4** for use in Question 2.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FA 2** to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 4** 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 the number of moles of potassium manganate(VII) present in the volume of **FA 2** calculated in (b).

moles of KMnO_4 = ... mol

- (ii) Use your answer to (i) and the equation at the top of page 2 to calculate the number of moles of hydrogen peroxide present in 25.0 cm³ of **FA 4**.

moles of H_2O_2 = ... mol

- (iii) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 4**.

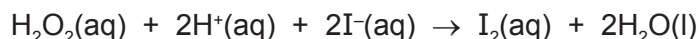
concentration of H_2O_2 in **FA 4** = ... mol dm⁻³

- (iv) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 1**.

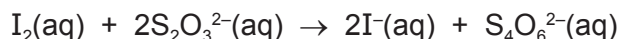
concentration of H_2O_2 in **FA 1** = ... mol dm⁻³
[4]

[Total: 12]

- 2 In **Question 1**, hydrogen peroxide was oxidised to form oxygen. Hydrogen peroxide can also be reduced to form water. This occurs, for example, when hydrogen peroxide, H_2O_2 , reacts with iodide ions, $\text{I}^-(\text{aq})$, to form iodine, $\text{I}_2(\text{aq})$.



The rate of this reaction can be measured by adding acidified hydrogen peroxide, H_2O_2 , to a mixture of iodide ions, I^- , thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, and starch indicator. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.



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

In this experiment you will measure two reaction times and use one of these to calculate the concentration of the thiosulfate ions in **FA 6**.

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

FA 4 is the diluted hydrogen peroxide, H_2O_2 , you prepared in **1(a)**.

FA 5 is 1.0 mol dm^{-3} potassium iodide, KI .

FA 6 is aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.
starch indicator

Read through the instructions carefully before starting any practical work.

(a) Method

- Use the measuring cylinder to transfer 20 cm^3 of **FA 3** into the 100 cm^3 beaker.
- Use the measuring cylinder to add 10 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Rinse the measuring cylinder with distilled water and drain.
- Use the measuring cylinder to transfer 20 cm^3 of **FA 5** into the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 20 cm^3 of **FA 6** into the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 10 cm^3 of starch indicator into the 250 cm^3 beaker.
- Add the contents of the 100 cm^3 beaker to the 250 cm^3 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.
- Rinse both beakers and drain.

Keep all FA solutions and starch indicator for use in (c).

reaction time = ... s [2]

(b) Calculations

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

- (i) The expression for the rate of reaction is shown.

$$\text{rate} = \frac{\text{concentration of iodine at the reaction time}}{\text{reaction time}}$$

The concentration of iodine at the reaction time is the concentration of iodine that would have been present at the time the blue-black colour appeared if no thiosulfate ions had been added.

In this experiment, you should assume the rate = $2.61 \times 10^{-5} \text{ mol dm}^{-3} \text{ s}^{-1}$.

Use this value for the rate to calculate the concentration of iodine that would have been present at the reaction time if no thiosulfate ions had been added.

$$\text{concentration of } \text{I}_2 = \dots\dots\dots \text{ mol dm}^{-3}$$

- (ii) Use your answer to (i) and the total reaction volume, to calculate the number of moles of iodine that would have been present at the reaction time if no thiosulfate ions had been added.

(If you were unable to calculate an answer to (i), you should use the value of $1.32 \times 10^{-3} \text{ mol dm}^{-3}$. This may not be the correct value.)

$$\text{moles of } \text{I}_2 = \dots\dots\dots \text{ mol}$$

- (iii) Calculate the number of moles of thiosulfate ions that reacted with the moles of I_2 , calculated in (ii).



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

- (iv) Use your answer to (iii) to calculate the concentration of thiosulfate ions in **FA 6**.

$$\text{concentration of } \text{S}_2\text{O}_3^{2-} \text{ in FA 6} = \dots\dots\dots \text{ mol dm}^{-3}$$

[4]

(c) Repeat the experiment in (a) using the following quantities of each reagent.

- add to the 100 cm³ beaker
20 cm³ of **FA 3**
10 cm³ of **FA 4**
- add to the 250 cm³ beaker
20 cm³ of **FA 5**
10 cm³ of **FA 6**
10 cm³ of starch indicator
10 cm³ of distilled water
- Add the contents of the 100 cm³ beaker to the 250 cm³ 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.

reaction time = ... s [1]

(d) (i) Explain the relationship between the value of the reaction time in (a) and the value of the reaction time in (c).

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

(ii) A student states that the error in the **total volume** of the reaction mixture in (a) is the same as the error in the **total volume** of the reaction mixture in (c).

State whether or not you agree with the student and explain your answer.

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

[4]

[Total: 11]

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) **FA 7, FA 8, FA 9** and **FA 10** are solutions that each contain a metal cation from those listed in the Qualitative Analysis Notes on page 10.

Carry out tests using reagents that will allow you to identify which metal ions are present.

Draw a single table to record your observations.

Complete the table below with your conclusions.

solution	FA 7	FA 8	FA 9	FA 10
metal ion present				

[11]

(b) Carry out the following test and record your observations.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 10 in a boiling tube add a 0.5 cm depth of 20 'vol' hydrogen peroxide, then add one drop of aqueous sodium hydroxide.	

[2]

(c) **FA 11** is a solid that contains one anion from those listed in the Qualitative Analysis Notes on page 11.

Place all of **FA 11** into a boiling tube and add a 3 cm depth of distilled water. Stir until all the solid has dissolved.

(i) Carry out the following test and record your observations.

<i>test</i>	<i>observations</i>
To a 1 cm depth of the solution of FA 11 in a test-tube add dilute sulfuric acid.	

(ii) Use your observations from (i) to identify the anion in **FA 11**.

The anion is

(iii) Use the remaining solution of **FA 11** in the boiling tube to carry out a further test to support your identification of the anion.
Record details of this test.

[4]

[Total: 17]

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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	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, $\text{SO}_3^{2-}(\text{aq})$	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

The Periodic Table of Elements

Group																											
1	2													13	14	15	16	17	18								
														1 H hydrogen 1.0								2 He helium 4.0					
																				Key							
																				atomic number atomic symbol name relative atomic mass							
3	4													5	6	7	8	9	10	11	12	13	14	15	16	17	18
Li lithium 6.9	Be beryllium 9.0													B boron 10.8	C carbon 12.0	N nitrogen 14.0	O oxygen 16.0	F fluorine 19.0	Ne neon 20.2								
11	12													13	14	15	16	17	18								
Na sodium 23.0	Mg magnesium 24.3													Al aluminium 27.0	Si silicon 28.1	P phosphorus 31.0	S sulfur 32.1	Cl chlorine 35.5	Ar argon 39.9								
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36										
K potassium 39.1	Ca calcium 40.1	Sc scandium 45.0	Ti titanium 47.9	V vanadium 50.9	Cr chromium 52.0	Mn manganese 54.9	Fe iron 55.8	Co cobalt 58.9	Ni nickel 58.7	Cu copper 63.5	Zn zinc 65.4	Ga gallium 69.7	Ge germanium 72.6	As arsenic 74.9	Se selenium 79.0	Br bromine 79.9	Kr krypton 83.8										
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54										
Rb rubidium 85.5	Sr strontium 87.6	Y yttrium 88.9	Zr zirconium 91.2	Nb niobium 92.9	Mo molybdenum 95.9	Tc technetium —	Ru ruthenium 101.1	Rh rhodium 102.9	Pd palladium 106.4	Ag silver 107.9	Cd cadmium 112.4	In indium 114.8	Sn tin 118.7	Sb antimony 121.8	Te tellurium 127.6	I iodine 126.9	Xe xenon 131.3										
55	56	57–71 lanthanoids	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86										
Cs caesium 132.9	Ba barium 137.3													Tl thallium 204.4	Pb lead 207.2	Bi bismuth 209.0	Po polonium —	At astatine —	Rn radon —								
87	88	89–103 actinoids	104	105	106	107	108	109	110	111	112			114	116												
Fr francium —	Ra radium —													Fl flerovium —	Lv livermorium —												

lanthanoids

actinoids

57	La	58	Ce	59	Pr	60	Nd	61	Pm	62	Sm	63	Eu	64	Gd	65	Tb	66	Dy	67	Ho	68	Er	69	Tm	70	Yb	71	Lu
lanthanum	cerium	praseodymium	neodymium	promethium	samarium	europium	gadolinium	terbium	dysprosium	holmium	erbium	thulium	ytterbium	lutetium															
138.9	140.1	140.9	144.4	—	150.4	152.0	157.3	158.9	162.5	164.9	167.3	168.9	173.1	175.0															
89	Ac	90	Th	91	Pa	92	U	93	Np	94	Pu	95	Am	96	Cm	97	Bk	98	Cf	99	Es	100	Fm	101	Md	102	No	103	Lr
	actinium		thorium		protactinium		uranium		neptunium		plutonium		americium		curium		berkelium		californium		einsteinium		fermium		mendelevium		nobelium		lawrencium
—		232.0	231.0	234.0	231.0	238.0	238.0	237.0	237.0	244.0	244.0	243.0	243.0	247.0	247.0	247.0	247.0	251.0	251.0	252.0	252.0	253.0	257.0	258.0	259.0	262.0	262.0	261.0	262.0

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9701/34

October/November 2016

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.
A copy of the Periodic Table is printed on page 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	
3	
Total	

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



- 1 You will find the relative atomic mass, A_r , of magnesium by measuring the volume of hydrogen produced when a known mass of metal reacts with an excess of acid.



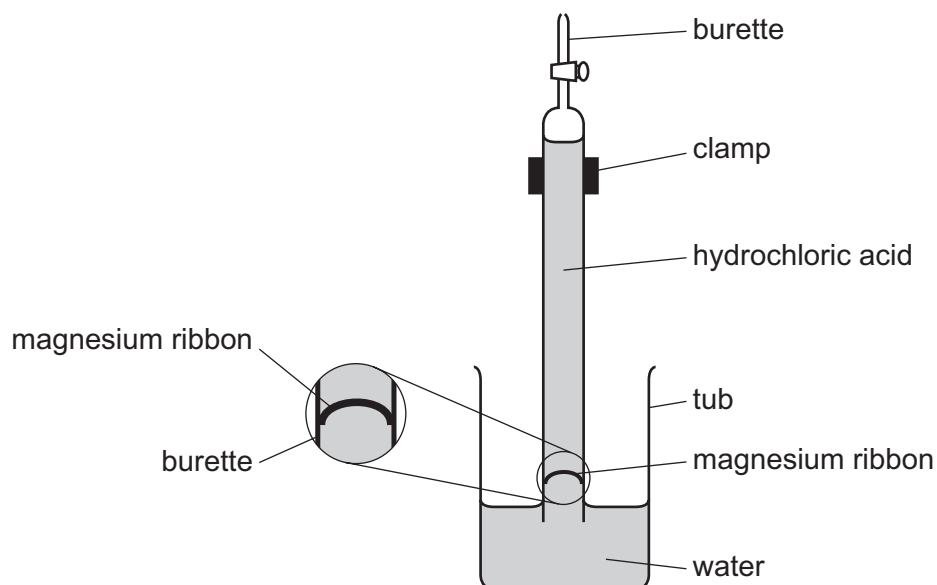
FB 1 is 1.00 mol dm^{-3} hydrochloric acid, HCl .

FB 2 is magnesium, Mg .

(a) Method

Read through the whole method before starting any practical work.

- Fill the tub with water to a depth of about 5 cm.
- Weigh the magnesium, **FB 2**, and note its mass below. If you are using a balance reading to 1 decimal place and the reading with the magnesium is zero, you should record this value.
- Fill the burette to about the 20 cm^3 mark with hydrochloric acid, **FB 1**.
- Add distilled water to reach the 0 cm^3 mark on the burette.
- Bend the magnesium strip into a U-shape.
- Place the magnesium in the burette so that it is above the liquid and friction holds it in position. Use a glass rod to push the magnesium about 2 cm into the burette.
- Hold a piece of paper towel over the open end of the burette, invert the burette and immediately place it in the tub of water. Remove the paper towel and clamp the burette as shown in the diagram.
- The liquid level should now be on the scale of the burette. If it is not, open the tap for a moment to allow the level to drop.



- Record the initial reading on the burette. Remember that the scale is now upside down.
- Leave the apparatus so that the acid from the burette diffuses around the magnesium and reacts.
- You should start **Question 2 or Question 3** while waiting for the reaction to complete.
- When all the magnesium has reacted, note and record the final reading on the burette.
- Calculate the volume of hydrogen produced.

Results

(b) 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 hydrogen produced.
(Assume that 1 mole of gas occupies 24.0 dm^3 under these conditions.)

moles of H_2 = ... mol

- (ii) Use your answer to (i) and the mass of magnesium used to calculate the A_r of magnesium.
(If you used a balance reading to 1 decimal place, you should assume that the mass of magnesium was 0.04 g correct to 2 decimal places.)

A_r of Mg = ...
[2]

- (c) (i) Calculate the percentage error in the mass and volume readings in this experiment.

- (ii) Suggest a change that could be made to reduce the greater error calculated in (i).

.....
.....
[3]

- (d) What would be the effect on the value of the A_r of magnesium calculated if the temperature of the room was much lower than that for your experiment? Explain your answer.

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

[Total: 10]

- 2 In **Question 1** you calculated the relative atomic mass, A_r , of magnesium by measuring the volume of hydrogen produced. The relative atomic mass can also be determined by investigating how much of the hydrochloric acid reacted with the magnesium.

The experiment described in **Question 1** was repeated, this time using 0.21 g of magnesium ribbon and 30.0 cm³ of 1.00 mol dm⁻³ hydrochloric acid. All the solution left in the burette and tub was kept and water added to make the total volume 250 cm³. This solution was labelled **FB 3**.

You will titrate **FB 3** using a known concentration of aqueous sodium carbonate to determine how much hydrochloric acid was left over after the reaction with magnesium.



FB 3 is the solution of hydrochloric acid described above.

FB 4 is aqueous sodium carbonate containing 2.64 g dm⁻³ Na₂CO₃.
bromophenol blue indicator

(a) Method

- Fill the burette with **FB 3**.
- Pipette 25.0 cm³ of **FB 4** into a conical flask.
- Add about 10 drops of bromophenol blue indicator.
- 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 consider necessary to obtain consistent results.
- Record, in a suitable form below, all of your burette readings and the volume of **FB 3** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FB 3** to be used in your calculations. Show clearly how you obtained this value.

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

(c) Calculations

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

- (i) Use the information on page 4 and the Periodic Table on page 12 to calculate the number of moles of sodium carbonate in the 25.0 cm³ of **FB 4** used in each titration.

moles of Na₂CO₃ = ... mol

- (ii) Use your answer to (i) to calculate the number of moles of hydrochloric acid present in the volume of **FB 3** recorded in (b).



moles of HCl present = ... mol

- (iii) Use your answer to (ii) to calculate the number of moles of hydrochloric acid present in 250 cm³ of **FB 3**.

moles of HCl present in 250 cm³ = ... mol

- (iv) Use the information on page 4 to calculate the number of moles of hydrochloric acid added to the magnesium.

moles of HCl added = ... mol

- (v) Calculate the number of moles of hydrochloric acid that reacted with the magnesium.

moles of HCl that reacted with the magnesium = ... mol

- (vi) Use your answer to (v) and the mass of magnesium used to calculate the relative atomic mass, A_r , of magnesium.



A_r of Mg = ...
[5]

- (d) A solution of sodium hydroxide was prepared at the same concentration, in mol dm^{-3} , as **FB 4**. A student repeated the titration but replaced **FB 4** with this solution of sodium hydroxide.

- (i) Explain the effect that replacing **FB 4** with this solution of sodium hydroxide would have on the volume of acid, **FB 3**, needed for the titration.

.....
.....

- (ii) If the sodium hydroxide had been stored for a long time it would not be suitable for use to find the concentration of the acid.

Suggest why storage for a long time would make the sodium hydroxide unsuitable.

.....
.....

[2]

[Total: 15]

3 Qualitative Analysis

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

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

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

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

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) (i) Half fill the 250 cm³ beaker with water. Heat the water to about 80 °C and then turn off the Bunsen burner. This is the hot water bath needed in the following tests.

To a 3–4 cm depth of aqueous silver nitrate in a test-tube, add a few drops of aqueous sodium hydroxide to give a grey / brown precipitate. Then add aqueous ammonia dropwise until the precipitate **just** disappears. This solution is Tollens' reagent and is needed in a following test.

FB 5, **FB 6** and **FB 7** are each known to be one of ethanol, propanal and propanone.

Carry out the following tests and complete the table.

<i>test</i>	<i>observations</i>		
	FB 5	FB 6	FB 7
To a 1 cm depth in a test-tube, add a few drops of acidified potassium manganate(VII) and place in the hot water bath.			
To a 0.5 cm depth in a test-tube, add a 1 cm depth of aqueous potassium iodide and a 1 cm depth of sodium chlorate(I). (This gives the same result as adding iodine and alkali.)			
To a few drops in a test-tube, add a 1 cm depth of Tollens' reagent and place in the hot water bath. Leave for several minutes.			

(ii) Use these observations to identify the unknown compounds.

FB 5 is

FB 6 is

FB 7 is

(iii) Choose another reagent that would give a similar result for propanal and propanone but a different result for ethanol.

Do not carry out this test.

reagent

result for propanal and propanone

result for ethanol

(iv) Choose another reagent that would give a similar result for ethanol and propanone but a different result for propanal.

Do not carry out this test.

reagent

result for ethanol and propanone

result for propanal

[8]

- (b) **FB 8** contains one cation and one anion from those listed on pages 10 and 11.
You are provided with solid **FB 8** and an aqueous solution of **FB 8**.

- (i) To a 1 cm depth of aqueous **FB 8** in a test-tube add a 1 cm depth of aqueous sodium hydroxide.

Keep the test-tube and contents for test (ii).

observation

- (ii) Transfer the contents of the test-tube from test (i) into a boiling tube and heat gently and **carefully**.

Allow to cool and keep the boiling tube and contents for test (iii).

observation

- (iii) Transfer a 1 cm depth of the mixture from test (ii) into a boiling tube and add a 2 cm depth of dilute hydrochloric acid. Heat gently and **carefully**.

observation

Allow to cool and keep the boiling tube and contents for test (iv).

- (iv) To the boiling tube from test (iii) add a piece of aluminium foil. Leave the boiling tube to stand.

observation

.....

- (v) Place a small spatula measure of solid **FB 8** in a hard-glass test-tube and heat it gently at first and then more strongly.

Identify **two** gases, other than water vapour, that are produced and give your evidence.

identity

evidence

identity

evidence

- (vi) From your observations in (i) to (v), write the formula of **FB 8**.

.....

- (vii) Write the **ionic** equation for the reaction that is occurring in test (i). Include state symbols.

.....

[7]

[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	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, $\text{SO}_3^{2-}(\text{aq})$	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

The Periodic Table of Elements

Group																											
1	2	Key														17	18										
		atomic number atomic symbol name relative atomic mass												1 H hydrogen 1.0													
3	4																										
Li lithium 6.9	Be beryllium 9.0																										
11	12																										
Na sodium 23.0	Mg magnesium 24.3																										
19	20																										
K potassium 39.1	Ca calcium 40.1																										
37	38																										
Rb rubidium 85.5	Sr strontium 87.6																										
55	56																										
Cs caesium 132.9	Ba barium 137.3																										
87	88																										
Fr francium —	Ra radium —																										

CANDIDATE
NAMECENTRE
NUMBER

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CANDIDATE
NUMBER

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CHEMISTRY

9701/35

Paper 3 Advanced Practical Skills 1

October/November 2016

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.
A copy of the Periodic Table is printed on page 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	
3	
Total	

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

- 1 In **Questions 1** and **2** you will determine the percentage purity of industrial grade calcium carbonate, CaCO_3 , by two different methods.

In the first method you will collect and measure the volume of gas given off in the reaction between a known mass of industrial grade calcium carbonate, in the form of small marble chips, and a known amount of dilute hydrochloric acid. The acid will be in excess. The impurities in the calcium carbonate will not react with the acid.



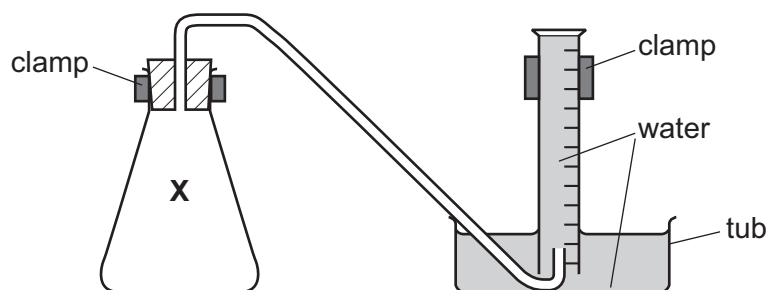
FA 1 is industrial grade calcium carbonate, CaCO_3 , in the form of small marble chips.

FA 2 is 2.00 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Read through the whole method before starting any practical work.

The diagram below may help you in setting up your apparatus.



- Fill the tub with water to a depth of about 5 cm.
- Fill the 250 cm^3 measuring cylinder **completely** with water. Hold a piece of paper towel firmly over the top, invert the measuring cylinder and place it in the water in the tub.
- Remove the paper towel and clamp the inverted measuring cylinder so the open end is in the water just above the base of the tub.
- Pipette 25.0 cm^3 of **FA 2** into the reaction flask labelled **X**.
- Check that the bung fits tightly in the neck of flask **X**, clamp flask **X** and place the end of the delivery tube into the inverted 250 cm^3 measuring cylinder.
- Weigh the container with **FA 1** and record the mass in the space on page 3.
- Remove the bung from the neck of the flask. Tip **FA 1** into the acid and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents. Swirl the flask occasionally until no more gas is evolved. Replace the flask in the clamp.
- Reweigh the container and any residue of **FA 1** and record the mass in the space on page 3.
- Calculate and record in the space on page 3 the mass of **FA 1** used.
- When no more gas is given off, measure and record the final volume of gas in the measuring cylinder in the space on page 3.

Keep the contents of flask X for use in Question 2.

Results

[2]

(b) 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 carbon dioxide gas collected in the measuring cylinder. (Assume that 1 mole of gas occupies 24.0 dm^3 under these conditions.)

moles of CO_2 = ... mol

- (ii) Use your answer to (i) and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO_3 = ... g

- (iii) Use your answer to (ii) and the mass of marble chips used in (a) to calculate a value for the percentage purity of the sample of industrial grade calcium carbonate, **FA 1**.

percentage purity of **FA 1** = ... %
[4]

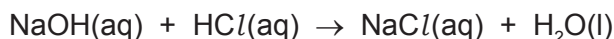
- (c) Not all the carbon dioxide given off in the reaction is collected in the measuring cylinder.

Suggest a change to the method which would lead to an increase in the volume of carbon dioxide collected.

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

[Total: 7]

- 2 You will determine the amount of hydrochloric acid remaining in flask **X** after the reaction with the marble chips in **Question 1**. You will do this by titration with sodium hydroxide of known concentration.



The impurities in the calcium carbonate will not react with the alkali.

FA 3 is 0.140 mol dm⁻³ sodium hydroxide, NaOH.
bromophenol blue indicator

(a) Method

- Transfer **all** the contents of flask **X** into the 250 cm³ volumetric flask.
- Rinse flask **X** with distilled water and add the washings to the volumetric flask. Add distilled water up to the mark.
- Stopper the volumetric flask and mix the contents thoroughly. Label this solution **FA 4**.
- Rinse the pipette then use it to transfer 25.0 cm³ of **FA 4** into a conical flask.
- Add about 10 drops of bromophenol blue indicator.
- Fill the burette with **FA 3**.
- 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.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 3** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b)** From your accurate titration results, obtain a suitable value for the volume of **FA 3** to be used in your calculations. Show clearly how you obtained this value.

25.0 cm³ of **FA 4** required cm³ of **FA 3**. [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 sodium hydroxide, NaOH, present in the volume of **FA 3** you calculated in **(b)**.

moles of NaOH = ... mol

- (ii) Use your answer to (i) and the equation on page 4 to determine the number of moles of hydrochloric acid, HCl, present in the 25.0 cm³ of **FA 4** pipetted in **(a)**.

moles of HCl = ... mol

- (iii) Use your answer to (ii) to calculate the number of moles of hydrochloric acid, HCl, remaining in flask **X** after the reaction in **1(a)**.

moles of HCl remaining = ... mol

- (iv) Use the relevant information on page 2 to calculate the number of moles of hydrochloric acid, HCl, pipetted into flask **X** in **1(a)**.

moles of HCl pipetted into flask **X** = ... mol

- (v) Use your answers to (iii) and (iv) to calculate the number of moles of hydrochloric acid, HCl, which reacted with the marble chips in flask **X**.

moles of HCl which reacted in flask **X** = ... mol

- (vi) Use your answer to (v), the equation in **Question 1** and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate, CaCO_3 , in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO_3 = ... g

- (vii) Use your answer to (vi) and the mass of marble chips recorded in **1(a)** to calculate the percentage purity of **FA 1**.

percentage purity of **FA 1** = ... %
[5]

- (d) You have carried out two different methods to find the percentage purity of industrial grade calcium carbonate.

A source of error in **Question 1** is that some carbon dioxide escapes before the bung can be inserted.

How would this affect the percentage purity of **FA 1** calculated in the two questions? Explain your answers.

Question 1

.....

.....

.....

Question 2

.....

.....

.....

[3]

[Total: 16]

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) **FA 5** and **FA 6** are solids each containing one cation and one anion.

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

<i>test</i>	<i>observations</i>	
	FA 5	FA 6
(i) Place a spatula measure of solid in a hard-glass test-tube and heat gently at first, then		
heat strongly until no further change takes place.		
Leave the tube to cool completely then add a 2 cm depth of dilute sulfuric acid to the solid residue. Shake the contents of the tube then leave it to stand.		

test	observations	
	FA 5	FA 6
(ii) Place a spatula measure of solid in a boiling tube and add a 2 cm depth of dilute sulfuric acid.		
Keep the solutions formed in (ii) for tests (iii) and (iv).		
(iii) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous sodium hydroxide.		
(iv) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous ammonia.		

- (v) Identify as many ions as you can from your observations. Write 'unknown' where you have not been able to identify an ion.

FA 5: cation anion

FA 6: cation anion

- (vi) Write an equation, including state symbols, for the reaction between **FA 6** and dilute sulfuric acid.

.....
[12]

(b) **FA 7** is a solution containing one anion from those listed on page 11. The anion is either a halide or contains nitrogen.

(i) You are to select suitable reagents to determine the identity of this anion. Record these in a suitable form below.

(ii) Use these reagents to carry out tests to identify the anion in **FA 7**.

Record your observations and conclusions in the space below.

[5]

[Total: 17]

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	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, $\text{SO}_3^{2-}(\text{aq})$	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

The Periodic Table of Elements

Group																									
1	2	Key												13	14	15	16	17	18						
		atomic number atomic symbol name relative atomic mass												1 H hydrogen 1.0											
3	4																								
Li lithium 6.9	Be beryllium 9.0																								
11	12																								
Na sodium 23.0	Mg magnesium 24.3																								
19	20																								
K potassium 39.1	Ca calcium 40.1																								
37	38																								
Rb rubidium 85.5	Sr strontium 87.6																								
55	56																								
Cs caesium 132.9	Ba barium 137.3																								
87	88																								
Fr francium —	Ra radium —																								

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9701/36

October/November 2016

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.
A copy of the Periodic Table is printed on page 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

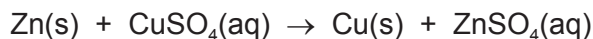
For Examiner's Use	
1	
2	
3	
Total	

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



- 1 Zinc metal is extracted from ores that, in addition to compounds of zinc, can also contain copper and silver. This means zinc metal is often impure.

You will investigate the percentage purity of a sample of zinc by reacting it with excess aqueous copper(II) sulfate and comparing the enthalpy change of the displacement reaction with the accepted value.



FB 1 is 1.00 mol dm⁻³ copper(II) sulfate, CuSO₄.

FB 2 is powdered impure zinc, Zn.

(a) Method

Read the whole method before starting any practical work.

- Weigh the container with **FB 2** and record the mass in the space below.
- Support the plastic cup in the 250 cm³ beaker.
- Use the measuring cylinder to transfer 25 cm³ of **FB 1** into the plastic cup.
- Place the thermometer in the solution and record the initial temperature in a suitable table of results. Tilt the cup if necessary so that the bulb of the thermometer is fully covered. This is the temperature at time zero ($t = 0$).
- Start timing and do not stop the clock until the whole experiment has been completed at $t = 8$ minutes.
- Measure and record the temperature of the **FB 1** in the cup every half minute up to and including $t = 2$ minutes.
- At $t = 2\frac{1}{2}$ minutes add all the **FB 2** into the cup and stir the contents until t is nearly 3 minutes.
- Measure and record the temperature of the mixture in the cup every half minute from $t = 3$ minutes until $t = 8$ minutes. Stir occasionally throughout this time.
- Weigh the container and any residual **FB 2**. Record this mass and calculate the mass of **FB 2** added.

Results

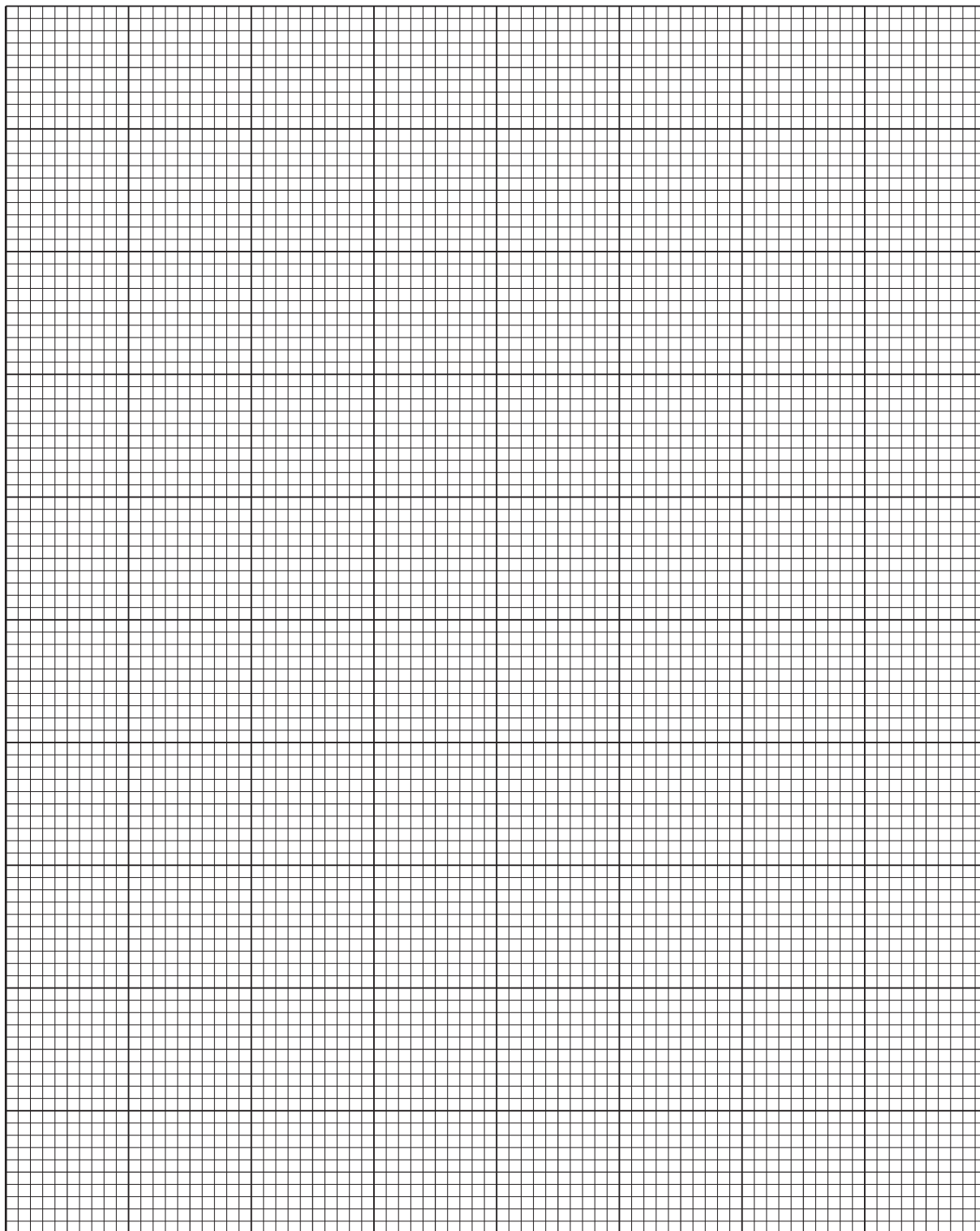
Mass

Temperature

I	
II	
III	
IV	

[4]

- (b) Plot a graph of temperature on the y-axis against time on the x-axis on the grid below. The scale for temperature should extend 3°C above your highest recorded temperature. You will use the graph to determine the theoretical maximum temperature rise at $t = 2\frac{1}{2}$ minutes.



I	
II	
III	
IV	

Draw two lines of best fit through the points on your graph, the first for the temperature before adding **FB 2** and the second for the temperature of the mixture after addition of **FB 2**. Extrapolate the lines to $t = 2\frac{1}{2}$ minutes and determine the theoretical maximum temperature rise, ΔT .

theoretical maximum temperature rise at $t = 2\frac{1}{2}$ minutes, $\Delta T = \dots\dots\dots^{\circ}\text{C}$ [4]

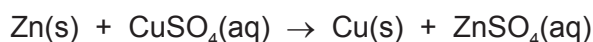
(c) Calculations

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

- (i) Use your answer to (b) to calculate the heat energy, in J, given out when **FB 2** was added to the **FB 1** in the cup.
(Assume that 4.2 J of heat energy raises the temperature of 1.0 cm³ of the mixture by 1.0 °C.)

heat energy given out = ... J

- (ii) Use your answer to (i) and the Periodic Table on page 12 to calculate the enthalpy change, in kJ mol⁻¹, for the displacement reaction.



You should assume that **FB 2** was pure zinc for this calculation.

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

[3]

- (d) The accepted value for the enthalpy change of this reaction is -217 kJ mol⁻¹.

Assuming no heat loss and that the other metals present in **FB 2** do not react with aqueous copper(II) sulfate, calculate the percentage of zinc present in **FB 2**.

percentage of Zn = ... % [1]

- (e) A student carried out the same experiment but used pieces of zinc instead of zinc powder. All quantities and the initial temperature of the aqueous copper(II) sulfate remained the same.

State and explain what effects this change would have on the graph plotted.

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

[Total: 14]

- 2 Solid hydrated copper(II) sulfate has the formula $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ where x is the number of moles of water of crystallisation present in 1 mole of compound.

You will determine the equation for the reaction that occurs when hydrated copper(II) sulfate is heated to remove the water of crystallisation producing anhydrous copper(II) sulfate.

FB 3 is hydrated copper(II) sulfate $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$.

(a) Method

Record all masses in the space below.

- Weigh the crucible and add 2.2–2.4 g of **FB 3**.
- Weigh the crucible plus **FB 3**.
- Place the crucible on the pipe-clay triangle and heat it gently for approximately 4 minutes.
- Leave the crucible to cool and reweigh the crucible plus residue.

Keep the crucible and residue for test (c).

I	
II	
III	
IV	

[4]

(b) Calculations

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

- (i) Calculate the mass of anhydrous copper(II) sulfate, CuSO_4 , produced after heating.

mass of CuSO_4 = ... g

- (ii) Calculate the mass of water lost by heating.

mass of water = ... g

- (iii) Use your answers to (i) and (ii) and the Periodic Table on page 12 to deduce the value of **x** in the formula $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$.

x is

- (iv) Use your answer to (iii) to complete the equation for the reaction that occurs when hydrated copper(II) sulfate is heated. You should include state symbols.



[4]

- (c) Place the cooled crucible, with the residue, **on a heatproof mat** and carefully add a few drops of water.

- (i) Note your observations.

.....

- (ii) Explain your observations in (i) in terms of the reaction occurring.

.....

[3]

- (d) Two students carried out the experiment in (a) and obtained values for **x** that did not agree with the accepted value. One student calculated a value that was less than the accepted value and the other student calculated a value that was more than the accepted value.

In each case, suggest a reason for the error and an improvement that could be made to minimise it. You can assume that the calculations were correctly carried out.

Value less than accepted value

error

improvement

Value more than accepted value

error

improvement

[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.

No additional tests for ions present should be attempted.

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

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

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

- (a) **FB 4** and **FB 5** are aqueous solutions of equal concentrations, in mol dm^{-3} . Each contains one anion and one cation. The cation is the same in both **FB 4** and **FB 5**.

Half fill the 250 cm^3 beaker with water. Heat the water to about 80°C and then turn off the Bunsen burner. This is the hot water bath needed in the tests below.

To about a 2 cm depth of aqueous silver nitrate in a test-tube, add a few drops of aqueous sodium hydroxide to give a grey/brown precipitate. Then add aqueous ammonia dropwise until the precipitate **just** disappears. This solution is Tollens' reagent and is needed in a test below.

- (i) Carry out the tests on separate samples of **FB 4** and **FB 5** and complete the table.

test	observations	
	FB 4	FB 5
To a 1 cm depth of solution in a test-tube in a test-tube rack, add a spatula measure of sodium carbonate.		
To a 1 cm depth of solution in a test-tube, add a few drops of acidified potassium manganate(VII). Place the test-tube in the hot water bath.		
To a 1 cm depth of Tollens' reagent in a test-tube, add a few drops of solution. Place the test-tube in the hot water bath and leave for several minutes.		

- (ii) From your observations in (i), identify the cation present in **both FB 4** and **FB 5**.

cation

- (iii) From your observations in (i), what can be deduced about the anion present in **FB 4**?

.....

- (iv) Place a 1 cm depth of **FB 4** and **FB 5** separately in two test-tubes.

Measure and record the temperature of the two solutions.

FB 4 °C **FB 5** °C

To each solution, add an approximately 2 cm length of magnesium ribbon. Measure and record the maximum temperature reached in each test-tube.

FB 4 + Mg °C **FB 5** + Mg °C

- (v) Explain why there is a difference in the temperature rise for the reactions of magnesium with solutions **FB 4** and **FB 5**.

.....

.....

.....

[8]

(b) **FB 6** is a solid that contains two cations from those listed on page 10.

You are to plan a series of experiments that will enable you to identify the cations present. You should then carry out your plan, record all the observations you made in a suitable table and identify the cations present.

cations present are and

[5]

[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	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, $\text{SO}_3^{2-}(\text{aq})$	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

The Periodic Table of Elements

Group																											
1	2	Key												13	14	15	16	17	18								
		atomic number atomic symbol name relative atomic mass												1 H hydrogen 1.0													
3	4																	5	6	7	8	9	10	11	12		
Li lithium 6.9	Be beryllium 9.0																	B boron 10.8	C carbon 12.0	N nitrogen 14.0	O oxygen 16.0	F fluorine 19.0					
11	12																	13	14	15	16	17	18				
Na sodium 23.0	Mg magnesium 24.3																	Al aluminium 27.0	Si silicon 28.1	P phosphorus 31.0	S sulfur 32.1	Cl chlorine 35.5	Ar argon 39.9				
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36										
K potassium 39.1	Ca calcium 40.1	Sc scandium 45.0	Ti titanium 47.9	V vanadium 50.9	Cr chromium 52.0	Mn manganese 54.9	Fe iron 55.8	Co cobalt 58.9	Ni nickel 58.7	Cu copper 63.5	Zn zinc 65.4	Ga gallium 69.7	Ge germanium 72.6	As arsenic 74.9	Se selenium 79.0	Br bromine 79.9	Kr krypton 83.8										
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54										
Rb rubidium 85.5	Sr strontium 87.6	Y yttrium 88.9	Zr zirconium 91.2	Nb niobium 92.9	Mo molybdenum 95.9	Tc technetium —	Ru ruthenium 101.1	Rh rhodium 102.9	Pd palladium 106.4	Ag silver 107.9	Cd cadmium 112.4	In indium 114.8	Sn tin 118.7	Sb antimony 121.8	Te tellurium 127.6	I iodine 126.9	Xe xenon 131.3										
55	56	57–71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86										
Cs caesium 132.9	Ba barium 137.3	lanthanoids	Hf hafnium 178.5	Ta tantalum 180.9	W tungsten 183.8	Re rhenium 186.2	Os osmium 190.2	Ir iridium 192.2	Pt platinum 195.1	Au gold 197.0	Hg mercury 200.6	Tl thallium 204.4	Pb lead 207.2	Bi bismuth 209.0	Po polonium —	At astatine —	Rn radon —										
87	88	89–103	104	105	106	107	108	109	110	111	112	114		116		—											
Fr francium —	Ra radium —	actinoids	Rf rutherfordium —	Db dubnium —	Sg seaborgium —	Bh bohrium —	Hs hassium —	Mt meitnerium —	Ds darmstadtium —	Rg roentgenium —	Cn copernicium —	Fl flerovium —		Lv livermorium —		—											

lanthanoids

actinoids

57	La	58	Ce	59	Pr	60	Nd	61	Pm	62	Sm	63	Eu	64	Gd	65	Tb	66	Dy	67	Ho	68	Er	69	Tm	70	Yb	71	Lu
	lanthanum		cerium		praseodymium		neodymium		promethium		samarium		euroium		gadolinium		terbium		dysprosium		holmium		erbium		thulium		ytterbium		lutetium
	138.9		140.1		140.9		144.4		—		150.4		152.0		157.3		158.9		162.5		164.9		167.3		168.9		173.1		175.0
89	Ac	90	Th	91	Pa	92	U	93	Np	94	Pu	95	Am	96	Cm	97	Bk	98	Cf	99	Es	100	Fm	101	Md	102	No	103	Lr
	actinium		thorium		protactinium		uranium		neptunium		plutonium		americium		curium		berkelium		californium		einsteinium		fermium		mendeleevium		nobelium		lawrencium
	—		232.0		231.0		238.0		—		—		—		—		—		—		—		—		—		—		—