CANDIDATE NAME	( )	CLASS	21S0		

RAFFLES INSTITUTION
2021 YEAR 6 PRELIMINARY EXAMINATION

Higher 2

CHEMISTRY 9729/04

Paper 4 Practical

25 August 2021

2 hours 30 minutes

Do NOT turn over the Question Booklet until you are told to do so.

READ THESE INSTRUCTIONS FIRST.

Write your name and class on the space provided when instructed to do so.
Give details of the practical shift and laboratory where appropriate, in the space provided.
Write in dark blue or black pen.
You may use a HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.

Answer all questions in the spaces provided on the Question Paper.
The number of marks is given in brackets [ ] at the end of each question or part question.

The use of an approved scientific calculator is expected, where appropriate.
You may lose marks if you do not show your working or if you do not use appropriate units.
Qualitative Analysis Notes are printed on pages 23 and 24.

Shift	
Laboratory	
Bench Number	

For Examiner's Use	
Question	**Marks**
1	**/ 16**
2	**/ 11**
3	**/ 14**
4	**/ 14**
Total	**/ 55**

This Question Paper consists of **23** printed pages and **1** blank page.

1 Determination of the solubility product of calcium iodate(V)

Calcium iodate(V), $Ca(IO_3)_2$, has low solubility in water. When a sample of this salt is mixed with water, a small amount dissolves and the following equilibrium is established in a saturated solution of $Ca(IO_3)_2$.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

You are to perform an experiment to determine the solubility product, K_{sp}, of $Ca(IO_3)_2(s)$.

When a sample of solid $Ca(IO_3)_2$ is added to a mixture containing specific volumes of aqueous potassium iodate(V), $KIO_3(aq)$, and aqueous potassium nitrate, $KNO_3(aq)$, both of known concentrations, a small amount of solid dissolves and the above equilibrium is established after some time.

The mixture is then filtered and the amount of iodate(V) ions, IO_3^-, in the filtrate is determined as described below.

- Excess potassium iodide is added to an acidified solution of the filtrate containing IO_3^- ions and iodine is liberated, as shown in equation 1.

  equation 1 $\quad IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \longrightarrow 3I_2(aq) + 3H_2O(l)$

- The liberated iodine is then titrated with a standard solution of sodium thiosulfate, as shown in equation 2.

  equation 2 $\quad 2S_2O_3^{2-}(aq) + I_2(aq) \longrightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$

(a) Preparation of a saturated solution of $Ca(IO_3)_2$ in $KIO_3(aq)$ and $KNO_3(aq)$, FA 1

You do not need to perform this part of the experiment.

1. Use a measuring cylinder to transfer 50.0 cm^3 of 0.0100 mol dm^{-3} $KIO_3(aq)$ to a clean and dry beaker.

2. Use another measuring cylinder to transfer 50.0 cm^3 of 0.0100 mol dm^{-3} $KNO_3(aq)$ to the beaker.

3. Add 3 spatulas of solid $Ca(IO_3)_2$ to the beaker.

4. Stir the mixture thoroughly. Leave the mixture to stand for several minutes to allow equilibrium to be reached.

5. Filter the mixture through a **dry** filter paper into a **dry** conical flask to collect the filtrate, **FA 1**, for titration in **(b)**.

FA 1 is the filtrate from **(a)**, a saturated solution of $Ca(IO_3)_2$ in KIO_3(aq) and KNO_3(aq).
FA 2 is 0.100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 3 is an aqueous solution of potassium iodide, KI.
FA 4 is 1.00 mol dm^{-3} dilute sulfuric acid, H_2SO_4.
Starch indicator

(b) Titration of filtrate, FA 1

1. Fill a burette with **FA 2**.
2. Pipette 25.0 cm^3 of **FA 1** into a 250 cm^3 conical flask.
3. Use a measuring cylinder to add about 10 cm^3 of **FA 3** to the conical flask.
4. Use another measuring cylinder to add about 10 cm^3 of **FA 4** to the conical flask.
5. Run **FA 2** from the burette into the conical flask until a pale yellow solution is obtained.
6. Add 5 drops of starch solution to the conical flask. Continue adding **FA 2** until the blue-black colour just disappears.
7. Record your titration results, to an appropriate level of precision, in the space provided below.
8. Perform sufficient titrations until consistent results are obtained.

(i) Titration results

[4]

(ii) From your titration results, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 2** = …….....

[1]

(c) Calculations

(i) Calculate the amount of $S_2O_3^{2-}$ ions present in the volume of **FA 2** recorded in **(b)(ii)**.

amount of $S_2O_3^{2-}$ ions = …….....
[1]

(ii) Calculate the amount of IO_3^- ions present in 25.0 cm^3 of the filtrate, **FA 1**.

amount of IO_3^- ions in 25.0 cm^3 of **FA 1** = …….....
[1]

(iii) Calculate the amount of IO_3^- ions present in the saturated solution prepared in **(a)**.

amount of IO_3^- ions in the saturated solution = …….....
[1]

(iv) Calculate the amount of IO_3^- ions from the $Ca(IO_3)_2$ that has dissolved.

amount of IO_3^- ions from the $Ca(IO_3)_2$ that has dissolved = …….....
[1]

(v) Calculate the amount of Ca^{2+} ions present in the saturated solution prepared in **(a)**.

amount of Ca^{2+} ions in the saturated solution = …….....
[1]

(vi) Write an expression for the solubility product, K_{sp}, of calcium iodate(V). Include units in your answer.

[1]

(vii) Calculate a value for the solubility product, K_{sp}, of calcium iodate(V).

K_{sp} = …….....
[1]

(d) In **(a)** step 5, **dry** filter paper and **dry** conical flask are used in the preparation of **FA 1**. Suggest and explain the likely consequence on the titre volume if these instructions were not followed.

…….

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…….

…….[1]

(e) A student claims that the K_{sp} of calcium iodate(V) is affected by the volume of KIO_3(aq). To investigate, the student repeated the procedure in **(a)** using four different volumes of KIO_3(aq) as shown in Table 1.1.

Table 1.1

Experiment	1	2	3	4
Volume of 0.0100 mol dm^{-3} KIO_3 / cm^3	0	25	75	100
Volume of 0.0100 mol dm^{-3} KNO_3 / cm^3	100	75	25	0

Titration of each collected filtrate was also carried out following the procedure in **(b)**.

(i) The total concentration of ions in a solution is known to affect the solubility product and the solubility of a salt. Hence, KNO_3(aq) is added to keep the total concentration of ions constant.

With the use of relevant calculations, show that the total concentration of ions prepared in Experiments 1 and 2 are the same.

[1]

(ii) Predict and explain how increasing the volume of $KIO_3(aq)$ used will affect the amount of IO_3^- ions obtained from the dissolved $Ca(IO_3)_2$ in **(a)**.

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……..[1]

(iii) State whether you agree with the student's claim that the K_{sp} of calcium iodate(V) is affected by the volume of $KIO_3(aq)$.

……..

Hence, sketch, on Figure 1.1, the graph of K_{sp} of calcium iodate(V) against volume of $KIO_3(aq)$ used you would expect to obtain.

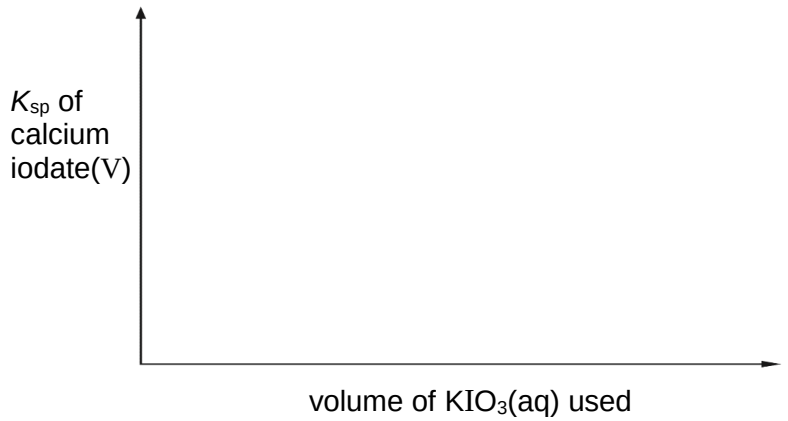

Figure 1.1

[1]

[Total: 16]

2 Planning

The **solubility** of potassium iodate(V), KIO_3, in water at a particular temperature is defined as:

the mass of potassium iodate(V) that will dissolve in and just saturate 100 g of water at that temperature.

When a solution of KIO_3 is cooled, it becomes saturated when crystals form in the solution.

(a) The solubility of KIO_3 in water increases with temperature. Explain if the dissolving of KIO_3 is exothermic or endothermic.

$$KIO_3(s) + aq \rightleftharpoons KIO_3(aq)$$

……

……

……[1]

A teacher carried out a series of experiments to investigate the solubility of KIO_3 at different temperatures. Experiment 1 was first carried out to determine the temperature at which 1 g of KIO_3 will dissolve in 4.0 cm^3 of deionised water.

Experiment 1

Step 1: Add 4.0 cm^3 of deionised water to a boiling tube containing 1.00 g solid KIO_3 at room temperature. The solid is undissolved despite stirring.

Step 2: Heat the boiling tube and its contents with stirring until all the solid is dissolved.

Step 3: Cool the mixture slowly while constantly stirring it. Record the temperature *T* when crystals first appear in the boiling tube.

The teacher recorded the temperature *T* as 80 °C, and concluded that 1.00 g of KIO_3 will dissolve in 4.0 cm^3 of water at 80 °C.

The same teacher then carried out Experiment 2 by continuing from Step 3 of Experiment 1.

Experiment 2

Step 4: Add a measured volume of deionised water to the mixture in the same boiling tube.

Step 5: Repeat the heating and cooling process in Steps 2 and 3.

The solubility of KIO_3 in water (in grams of solid per 100 g of water) can then be calculated using the formula below.

$$\text{solubility} = \frac{100}{\text{volume of water}} \times \text{mass of } KIO_3 \text{ dissolved}$$

where the volume of water is measured in cm^3.

You may assume that the density of water is 1.00 g cm^{-3} and remains constant over the temperature range in this experiment.

(b) Using the given formula and the teacher's experimental results, calculate the solubility of KIO_3 at 80 °C, in grams per 100 g of water.

[1]

(c) Given that the solubility of KIO_3 at 10 °C is 6.25 g per 100 g of water, calculate the volume of water that will just dissolve 1.00 g of KIO_3 at 10 °C.

[1]

(d) Plan a procedure to collect sufficient data to allow a graph of the solubility of KIO_3 against temperature to be drawn. Your plan should make use of the same procedure as described in Experiments 1 and 2, and should also allow you to determine the solubility of KIO_3 at about 10 °C and 80 °C.

Your experiment should begin with the same quantities as described in Step 1.

You may assume that you are provided with

- weighing bottle containing about 1 g KIO_3,
- deionised water,
- boiling tube,
- ice cubes,
- thermometer covering the temperature range 0 °C to 100 °C,
- the equipment normally found in a school or college laboratory.

In your plan you should include brief details of

- the apparatus you would use,
- the quantities you would use,
- the procedure you would follow and the measurements you would make,
- practical details of how you would
  - decide at which point the temperature of the mixture is to be taken,
  - make adjustments to the experiment, particularly when no crystals appear even after reaching room temperature,
  - ensure a wide range of results suitable for the graph.

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[6]

(e) Draw tables with appropriate headings to show the data you would record when carrying out your experiments, and show how you would use your data to calculate the solubility at each temperature.

[2]

[Total: 11]

3 Determination of the composition of a mixture

FA 4 is 1.00 mol dm^{-3} sulfuric acid.
FA 5 is a mixture of a metal, **M**, and magnesium oxide, MgO.

When **FA 5** is added to a known excess of sulfuric acid, both the metal, **M**, and magnesium oxide will react with sulfuric acid in an exothermic reaction. The temperature change of this reaction can be used to determine the percentage by mass of magnesium oxide in the mixture.

(a) Determination of the heat change in reacting a mixture with sulfuric acid

In this experiment, you will measure the temperature of the contents of a polystyrene cup at timed intervals, before and after **FA 5** is added. You will analyse your results graphically in order to determine an accurate value for the temperature change of the mixture, caused by **FA 5** reacting with **FA 4**.

You will use this value to calculate the heat change, *q*, in reacting **FA 5** with **FA 4**.

In an appropriate format in the space provided on page 14, prepare tables in which to record results for your experiment in **(a)**:

- all weighings to an appropriate level of precision,
- all values of temperature, *T*, to an appropriate level of precision,
- all values of time, *t*, recorded to the nearest 0.5 min.

It is important that you measure each temperature at the specified time.

1. Weigh the capped bottle containing **FA 5**.

2. Place one polystyrene cup inside a second polystyrene cup. Place these in a glass beaker to prevent them from tipping over.

3. Use a measuring cylinder to transfer 50 cm^3 of **FA 4** into the first polystyrene cup and put the lid on.

4. Insert the thermometer through the lid and use it to stir the **FA 4** in the cup. Read and record its temperature, *T* (time, *t* = 0.0 min).

5. Continue to stir the **FA 4**. Read and record *T* every minute.

6. At **exactly** three minutes, open the lid and transfer all the **FA 5** to the polystyrene cup. Replace the lid quickly and stir the mixture but do not read *T*.

7. Continue to stir the mixture. Read and record *T* at *t* = 3.5 min.

8. Continue to stir the mixture. Read and record *T* at *t* = 4.0 min and every minute until *t* = 9.0 min.

9. Reweigh the emptied bottle and its cap.

(i) Results

[4]

(ii) Plot a graph of temperature, T, on the y-axis, against time, t, on the x-axis, on the grid in Fig. 3.1.

Draw a best-fit straight line taking into account all of the points before t = 3.0 min.

Draw another best-fit straight line taking into account all of the points after the temperature of the mixture has started to fall.

Extrapolate (extend) both lines to t = 3.0 min.

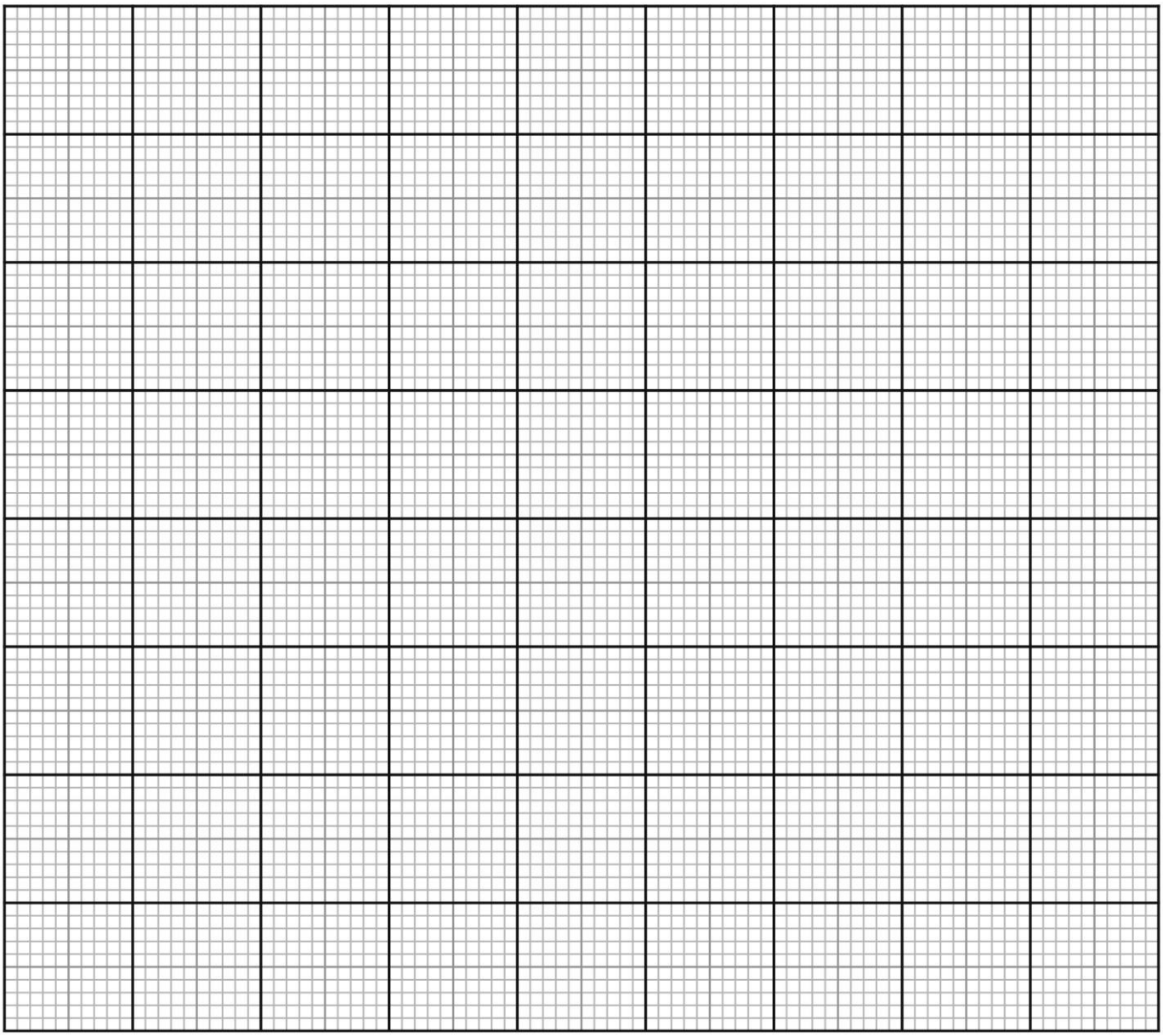

Fig. 3.1

[3]

(iii) From your graph, read the minimum temperature, T_{min}, and the maximum temperature, T_{max}, at t = 3.0 min. Record these values in the spaces provided.

Determine the temperature change, ΔT, at t = 3.0 min.

T_{min} = ……..

T_{max} = ……..

ΔT = ……..

[1]

(iv) Calculate the heat change, q, for your experiment using the value you determined in **(a)(iii)**.

You should assume that the specific heat capacity of the solution is 4.18 J g^{-1} K^{-1}, and that the density of the solution is 1.00 g cm^{-3}.

q = ……..

[1]

(v) Suggest the effect that using 100 cm^3, rather than 50 cm^3, of **FA 4** would have on your value for ΔT. Explain the effect this will have on the value for q.

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……..……..

[1]

(b) The metal, **M**, present in **FA 5** is one of the metal ions in the Qualitative Analysis Notes on pages 23-24.

(i) Suggest a reason based on your experiment in **(a)** why **M** cannot be calcium.

……..…....

……..…....

..……..…..

[1]

(ii) A small amount of **M** was added to sulfuric acid and the resultant solution was neutralised using sodium hydroxide. When excess sodium hydroxide was added, a white precipitate was observed.

State the identity of **M**.

M is ……..…. [1]

(c) Table 3.1 gives the values of enthalpy change of reaction, ΔH_r, for the reaction between some metals and sulfuric acid.

Assuming that the percentage by mass of magnesium oxide in **FA 5** was 87 %, use the identity of **M** from **(b)(ii)** and its respective ΔH_r value from Table 3.1 to determine the enthalpy change of reaction, ΔH_1, for the reaction between magnesium oxide and sulfuric acid.

Table 3.1

substance	ΔH_r / kJ mol^{-1}
aluminium	−531.6
magnesium	−466.8
zinc	−153.9

[A_r: A*l*, 27.0; Mg, 24.3; Zn, 65.4; O, 16.0]

ΔH_1 = ……..…. [2]

[Total: 14]

4 Qualitative Analysis

(a) **FA 6** is an aqueous solution containing a sodium salt of the polyatomic anion, $\mathbf{R^{2-}}$.

In aqueous solution, $\mathbf{R^{2-}}$ reacts very slowly with water to produce anion **P**, cation **Q** and oxygen gas.

equation 3 $\quad 2H_2O(l) + 2\mathbf{R^{2-}}(aq) \rightarrow 4\mathbf{P}(aq) + 4\mathbf{Q}(aq) + O_2(g)$

Perform the tests described in Table 4.1, and record your observations in the table. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

Table 4.1

	test	observations
1	Test the solution of **FA 6** with Universal Indicator Paper.	
2	Add 1 cm depth of **FA 6** to a boiling tube. Add aqueous sodium hydroxide until no further change occurs, then warm the boiling tube gently.	
	Allow the mixture to cool slightly. Add 1 piece of aluminium foil, then warm the boiling tube gently.	
3	Add 1 cm depth of **FA 6** to a test-tube. Add 1 cm depth of aqueous barium nitrate. Add dilute nitric acid dropwise until no further change occurs.	

[3]

(b) **(i)** In Table 4.2, identify anion **P** and cation **Q,** and quote evidence from **(a)** to support your answers.

Table 4.2

	identity	evidence
anion **P**		
cation **Q**		

[2]

(ii) Hence, use equation 3 to deduce the identity of $\mathbf{R^{2-}}$.

$\mathbf{R^{2-}}$ is ……[1]

(c) **FA 7** contains a cation from the Qualitative Analysis Notes on pages 23-24.

(i) To 1 cm depth of **FA 7** in a test-tube, add 1 cm depth of sodium carbonate. Record your observations. Test and identify any gases evolved.

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[2]

(ii) Deduce the identity of the cation in **FA 7**. In your answer, you should identify the species responsible for the observations.

……...

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……...
[1]

(d) The following steps are performed to investigate the reaction between **FA 6** and potassium iodide, and the role of **FA 7** in the reaction.

Carry out the following steps.

1. Label two test tubes A and B.
2. Add 2 cm depth of **FA 6** each to test-tubes A and B.
3. Add 1 drop of **FA 7** to test-tube A, but not to test-tube B.
4. Place test-tubes A and B in a 250 cm^3 beaker, positioned on a white tile.
5. In two separate 10 cm^3 measuring cylinders, measure out 3.0 cm^3 of potassium iodide each.
6. Transfer, at the same time, 3.0 cm^3 of the measured potassium iodide into test-tubes A and B.
7. Observe the changes in test-tubes A and B for 5 minutes.

   Keep the contents of test-tubes A and B for (iii).

(i) Describe one major difference in the observations of test-tubes A and B from steps 6 to 7.

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……...
[1]

(ii) Deduce the role of **FA 6** in the reaction and explain your answer with reference to the observations in **(i)**.

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[1]

(iii) To test-tubes A and B, add aqueous sodium hydroxide until no further change occurs. Record your observations in Table 4.3.

Table 4.3

test-tube A	test-tube B

[1]

(iv) Deduce the role of **FA 7** in this reaction, quoting evidence from parts **(i)** and **(iii)** to support your answer.

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[2]

[Total: 14]

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Qualitative Analysis Notes
[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	***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)	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

(b) Reactions of anions

anion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq))
nitrate, NO_3^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acids)
sulfite, SO_3^{2-}(aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in dilute strong acids)

(c) Tests for gases

gas	*test and test result*
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 aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

halogen	*colour of element*	*colour in aqueous solution*	*colour in hexane*
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I_2	black solid / purple gas	brown	purple