YISHUN INNOVA JUNIOR COLLEGE
JC 2 PRELIMINARY EXAMINATION
Higher 2

/

CANDIDATE NAME	

CLASS		DATE	

H2 CHEMISTRY

9729/04

Paper 4
Practical Paper

/

Candidates answer on question paper.
No Additional Materials are required

Tuesday

24 August 2021
2 hours 30 minutes

READ THESE INSTRUCTIONS FIRST

Write your name and class in the spaces at the top of this page.
Give details of the practical shift 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.

Answer **all** questions in the spaces provided on the Question Paper.
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 19 and 20.
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.

Shift
Laboratory

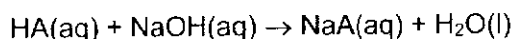| For Examiner's use	
1	19
2	7
3	17
4	12
Total	55

This document consists of **19** printed pages and **1** blank page.

[Turn over

Answer all the questions in the spaces provided.

1   **To determine the enthalpy change of neutralisation, ΔH_n**

The enthalpy change of neutralisation between an acid and an alkali can be determined using a type of experiment known as thermometric titration. One way of performing a thermometric titration involves using a fixed volume of alkali with progressive addition of small volumes of acid and monitoring the temperature of the reaction mixture during the process.

You are to determine the enthalpy change of neutralisation for the reaction given below.

$$HA(aq) + NaOH(aq) \rightarrow NaA(aq) + H_2O(l)$$

FA 1 is 1.80 mol dm^{-3} HA
FA 2 is aqueous sodium hydroxide, NaOH

In this experiment, you will measure the temperature of the contents of a polystyrene cup. You will analyse your results graphically in order to determine an accurate value for the temperature change of the mixture, caused by the neutralisation of aqueous HA and NaOH.

You will use this value to calculate the heat change, q, for the experiment and hence determine a value for the enthalpy change of neutralisation for the reaction, ΔH_n.

(a)   Procedure

1. Place one polystyrene cup in a 250 cm^3 beaker.

2. Rinse and fill the burette with **FA 1**.

3. Use a measuring cylinder to transfer 25 cm^3 of **FA 2** into the polystyrene cup.

4. Stir the solution in the cup with the thermometer. Read and record its temperature.

5. Run 5.00 cm^3 of **FA 1** into the cup. Stir the solution with the thermometer. Record the new temperature and volume of **FA 1** added.

6. Run a second portion of 5.00 cm^3 of **FA 1** into the cup. Stir and record the new temperature and the total volume of **FA 1** added.

7. Continue adding **FA 1** in 5.00 cm^3 portions. Stir and record each new temperature and the total volume of **FA 1** until a total of 45.00 cm^3 has been added.

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Results

In an appropriate format in the space below, prepare a table to record the results of your experiment:

- all measurements of volume used
- all values of temperature, T, to an appropriate level of precision

Table of results

[4]

(b) Plot a graph of temperature, T, on the y-axis, against total volume of **FA 1** added, on the x-axis. The temperature axis should allow you to include a point at least 2 °C greater than the maximum temperature recorded.

Draw a best-fit straight line taking into account all of the points for the increase in temperature of the mixture. Draw another best-fit straight line taking into account all of the points for the cooling of the solution.

Extrapolate the two lines and determine the
- maximum temperature reached, T_{max},
- maximum temperature increase, ΔT_{max},
- corresponding volume of **FA 1** added.

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T_{max} = ...

ΔT_{max} = ...
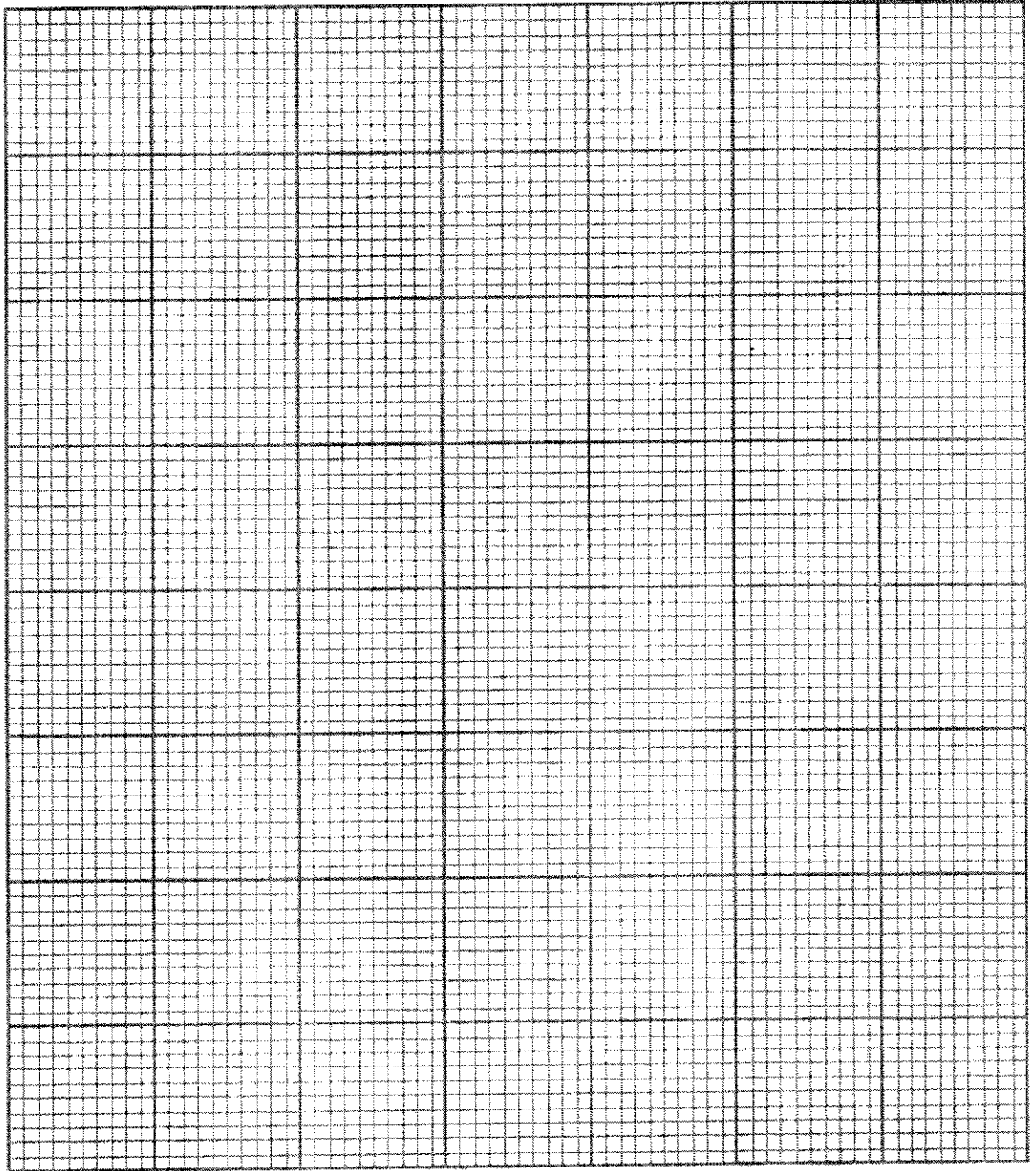
Volume of **FA 1** added = ...

[4]

(c) Explain the shape of the graphs before and after the maximum temperature is reached.

before maximum temperature is reached

....

....

after maximum temperature is reached

....

....

[2]

(d) **(i)** Calculate the number of moles of HA present in the volume of **FA 1** recorded in **(b)**.

number of moles of HA = ...

[1]

(ii) Using your answers to **(b)**, calculate the heat change, q, when **FA 1** has completely neutralised 25 cm^3 of sodium hydroxide.

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 = ...

[2]

(iii) Determine the enthalpy change of neutralisation, ΔH_n, for the reaction.

ΔH_n = ...
[1]

(e) The maximum error in a thermometer reading is ±0.1 °C.
Calculate the maximum percentage error in the increase in temperature recorded.

maximum percentage error = ...
[1]

(f) Apart from using a thermometer with a greater level of precision, suggest one improvement that could be made to the method carried out in (a).

....

....
[1]

(g) A student decided to perform the same experiment in (a) but used aqueous ammonia instead of aqueous sodium hydroxide.

Suggest what effect, if any, would replacing aqueous sodium hydroxide with aqueous ammonia have on the value of enthalpy change of neutralisation calculated in d(iii).

effect ....

explanation ....

....
[1]

(h) Another student repeated the same experiment in **(a)** but used **FB 1** which is 0.9 mol dm^{-3} of HA instead of **FA 1**.

Suggest what effect, if any, would replacing **FA 1** with **FB 1** have on the maximum temperature increase, ΔT_{max}.

effect ....

explanation ....

....

[2]

[Total:19]

2 Qualitative analysis

FA 3 contains one cation and two anions
FA 4 contains two cations

Carry out the following tests. Carefully record your observations in **Table 2.1.**

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved.

Table 2.1

		Test	Observations
(a)		Place a spatula of **FA 3** into a test tube, then add approximately 1 cm³ of dilute nitric acid.	
(b)		Place a spatula of **FA 3** into a boiling tube, then add about 15 cm³ of distilled water. Stopper and shake to mix the solution. Use a 1 cm depth of the **FA 3** solution obtained in a test-tube for each of tests **(i) to (iv).**	
	(i)	Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.	
	(ii)	Add aqueous ammonia slowly, with shaking, until no further change is seen.	
	(iii)	Add 10 drops of aqueous barium chloride, then add dilute nitric acid, with shaking, until in excess.	
	(iv)	Add a few drops of aqueous silver nitrate. Decant, then add aqueous ammonia until in excess.	

[3]

(c) Identify the cation present in **FA 3**. Use evidence from your observations in **Table 2.1** to support your deduction.

Cation in **FA 3**: ....

Evidence: ....

....

....
[1]

(d) Identify the anions present in **FA 3**.

Anions in **FA 3**: .... and ....
[1]

(e) FA 4 contains two cations.

A student added aqueous ammonia to a sample of **FA 4** and observed the formation of a green precipitate that was insoluble in excess of the reagent. He concluded that iron(II) ions were present.

Next, he placed a spatula of **FA 4** in a boiling tube and heated the solid gently. The gas evolved turned moist red litmus blue. Upon cooling, a brown residue was obtained.

(i) Using **Table 2.2**, plan and carry out one other test that will enable you to identify the other cation in **FA 4**.

You should start by dissolving half a spatula of **FA 4** in approximately 10 cm³ of distilled water. Use this solution of **FA 4** in your test.

Describe your test briefly and state your observations.

Table 2.2

Test	Observations

[1]

(ii) Identify the other cation in **FA 4** and write an ionic equation for the observation in **e(i)** that allows you to confirm its identity.
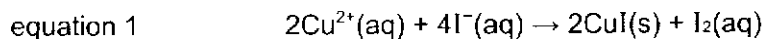
The other cation in **FA 4**: ....

Ionic equation: ....
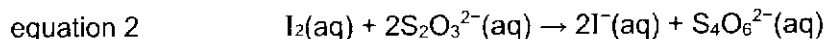[1]

[Total:7]

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3   **Determination of the water of crystallisation in hydrated copper(II) sulfate**

The addition of an excess of potassium iodide, KI, to a solution of Cu^{2+} ions produces iodine, I_2, and a stable precipitate of CuI. The I_2 turns the solution brown.

equation 1 $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$

The iodine produced can be titrated with a standard solution of $Na_2S_2O_3$ as shown in equation 2.

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

FA 8 is an aqueous solution made by dissolving 32.5 g of $CuSO_4.xH_2O$ in 1.00 dm^3 of solution.

You are also provided with
FA 5, 0.150 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$,
FA 6, dilute sulfuric acid, H_2SO_4,
FA 7, 1.00 mol dm^{-3} potassium iodide, KI,
starch indicator.

In this experiment you will determine the value of **x** in the formula for hydrated copper(II) sulfate, $CuSO_4.xH_2O$. You will prepare a mixture containing **FA 6**, **FA 7** and **FA 8** and titrate the iodine produced against **FA 5**.

(a)   (i)   Titration of a mixture against FA 5

1. Fill a burette with **FA 5**.

2. Use a pipette to transfer 25.0 cm^3 of **FA 8** into a 250 cm^3 conical flask.

3. Use a measuring cylinder to add 10 cm^3 of **FA 6** to the conical flask from step 2.

4. Use a measuring cylinder to add 10 cm^3 of **FA 7** to the same conical flask from step 3. A white precipitate forms in a brown solution.

5. Run **FA 5** from the burette into the flask. Near the end-point, when the brown solution becomes pale, add about 20 drops of the starch indicator.

6. Continue adding **FA 5** slowly. The end-point is reached when the **solution** first becomes colourless. The white precipitate remains.

7. Record your titration results, to an appropriate level of precision, in the space provided on Page 13.

8. Discard the contents of the conical flask **immediately** down the sink. Wash out the conical flask thoroughly with tap water.

9. Repeat points 2 to 8 until consistent results are obtained.

Titration results

[6]

(ii) From your titrations, obtain a suitable volume of **FA 5** to be used in your calculations. Show clearly how you obtained this volume.

Volume of **FA 5** = ...

[1]

(b) **(i)** Calculate the amount of copper(II) ions, Cu^{2+}, in 25.0cm³ of **FA 8**.

amount of Cu^{2+} in **FA 8** = ...

[1]

(ii) Calculate the concentration of copper(II) ions, $[Cu^{2+}]$, of **FA 8**.

concentration of Cu^{2+} in **FA 8** = ...

[1]

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(iii) Use your answer from **(b)(ii)** to calculate the M_r of hydrated copper(II) sulfate.

M_r of hydrated copper(II) sulfate = ...

Hence, deduce the value of **x**.
Show your working.
[A_r: H, 1.0; O, 16.0; S, 32.1; Cu, 63.5]

x = ...

[5]

(c) Identify two different chemical processes that use iodide ions in this experiment.

....

....

[1]

(d) A student suggests that the experiment could be made more accurate if the volume of **FA 7** was measured using a burette.

(i) Give a reason why the student might make this suggestion.

....

....

[1]

(ii) Explain whether this change will improve the accuracy of the experiment.

....

....
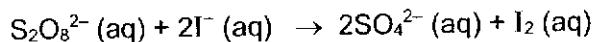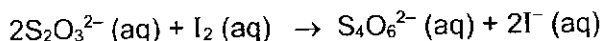
[1]

[Total: 17]

4 Planning

The method of initial rates is a commonly used technique for deriving rate laws. To determine the initial rate of a reaction, we can time how long it takes to reach an identifiable point early in the reaction.

In solution, iodide ions, I^-, are oxidised by peroxodisulfate ions, $S_2O_8^{2-}$.

$$S_2O_8^{2-} \text{ (aq)} + 2I^- \text{ (aq)} \rightarrow 2SO_4^{2-} \text{ (aq)} + I_2 \text{ (aq)}$$

If sodium thiosulfate and starch are added to the reaction mixture, the blue-black colour of an iodine-starch complex appears suddenly after some time. This occurs when all of the thiosulfate ions, $S_2O_3^{2-}$, present in the mixture have reacted with the iodine formed in the reaction above. This is the identifiable point in the reaction.

$$2S_2O_3^{2-} \text{ (aq)} + I_2 \text{ (aq)} \rightarrow S_4O_6^{2-} \text{ (aq)} + 2I^- \text{ (aq)}$$

You are to plan an experiment to investigate how the rate of reaction between potassium peroxodisulfate and potassium iodide depends on the concentration of potassium peroxodisulfate based on a graphical analysis.

A preliminary experiment, using approximate volumes of solution, indicates that the time taken for the iodine-starch complex to form doubles when the potassium peroxodisulfate is diluted with an equal amount of water.

(a) Using the results of the preliminary experiment, predict the order of reaction with respect to potassium peroxodisulfate. Explain your answer.

....

....

....

....

[2]

(b) Explain why it is important that the iodine formed by oxidation reacts with the sodium thiosulfate and is converted back to iodide ions.

....

....

....

[1]

(c) Explain why the volume of aqueous sodium thiosulfate should be measured precisely, using a burette, in the experiment.

....

....

....

[1]

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(d) Consider the description of the experiment given in the previous page.

Write a plan for a series of experiments to investigate your prediction in **(a)**.

You may assume that you are provided with:

- 0.60 mol dm^{-3} potassium iodide
- 0.20 mol dm^{-3} potassium peroxodisulfate
- 0.01 mol dm^{-3} sodium thiosulfate
- distilled water
- starch indicator solution
- the equipment normally found in a school laboratory

A first experiment is carried out using the following quantities.

- 20 cm^3 potassium iodide
- 40 cm^3 potassium peroxodisulfate
- 20 cm^3 sodium thiosulfate
- 0 cm^3 distilled water
- 10 cm^3 starch indicator solution

Give a step-by-step description of the method you would use in further experiments.

Your plan should contain the following:

- the apparatus you would use,
- a table to indicate the different quantities of reagents you would use,
- the procedure you would follow,
- the measurements you would make to allow a suitable graph to be drawn in order to investigate your prediction in **(a)**.

....

....

....

....

....

....

....

....

....

....

....

....

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[6]

(e) Assuming that your prediction in **(a)** is correct, sketch on Fig. 4.1 the graph you would expect to obtain from your results. Label the axes.
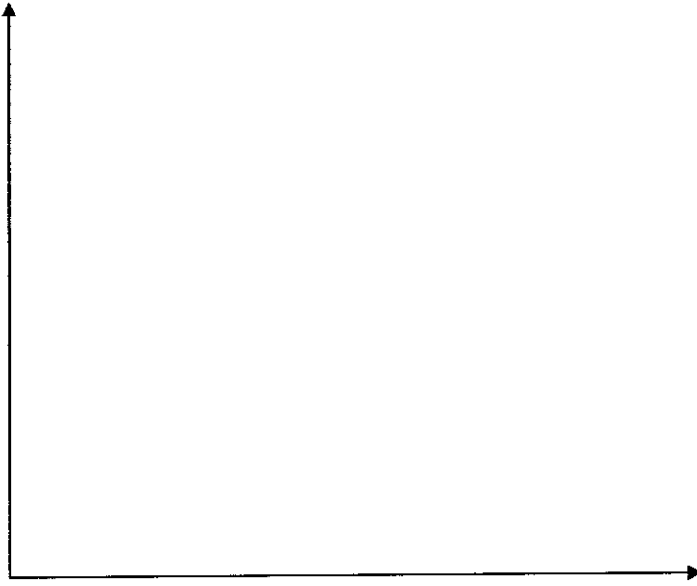

Fig. 4.1

[2]

[Total: 12]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	reaction with	
	NaOH(aq)	NH₃(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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

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(b) Reactions of anions

ions	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 → (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) Test for gases

ions	reaction
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

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Practical Paper

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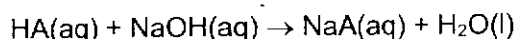The number of marks is given in brackets [ ] at the end of each question or part question.

Shift
Laboratory

For Examiner's use	
1	19
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4	12
Total	55

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[Turn over

Answer **all** the questions in the spaces provided.

1   To determine the enthalpy change of neutralisation, ΔH_n

The enthalpy change of neutralisation between an acid and an alkali can be determined using a type of experiment known as thermometric titration. One way of performing a thermometric titration involves using a fixed volume of alkali with progressive addition of small volumes of acid and monitoring the temperature of the reaction mixture during the process.

You are to determine the enthalpy change of neutralisation for the reaction given below.

$$HA(aq) + NaOH(aq) \rightarrow NaA(aq) + H_2O(l)$$

FA 1 is 1.80 mol dm^{-3} HA
FA 2 is aqueous sodium hydroxide, NaOH

In this experiment, you will measure the temperature of the contents of a polystyrene cup. You will analyse your results graphically in order to determine an accurate value for the temperature change of the mixture, caused by the neutralisation of aqueous HA and NaOH.

You will use this value to calculate the heat change, q, for the experiment and hence determine a value for the enthalpy change of neutralisation for the reaction, ΔH_n.

(a)   Procedure

1. Place one polystyrene cup in a 250 cm^3 beaker.

2. Rinse and fill the burette with **FA 1**.

3. Use a measuring cylinder to transfer 25 cm^3 of **FA 2** into the polystyrene cup.

4. Stir the solution in the cup with the thermometer. Read and record its temperature.

5. Run 5.00 cm^3 of **FA 1** into the cup. Stir the solution with the thermometer. Record the new temperature and volume of **FA 1** added.

6. Run a second portion of 5.00 cm^3 of **FA 1** into the cup. Stir and record the new temperature and the total volume of **FA 1** added.

7. Continue adding **FA 1** in 5.00 cm^3 portions. Stir and record each new temperature and the total volume of **FA 1** until a total of 45.00 cm^3 has been added.

Results
In an appropriate format in the space ▮▮▮▮, prepare a table to record the results of your experiment:

- all measurements of volume used
- all values of temperature, T, to an appropriate level of precision

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Table of results

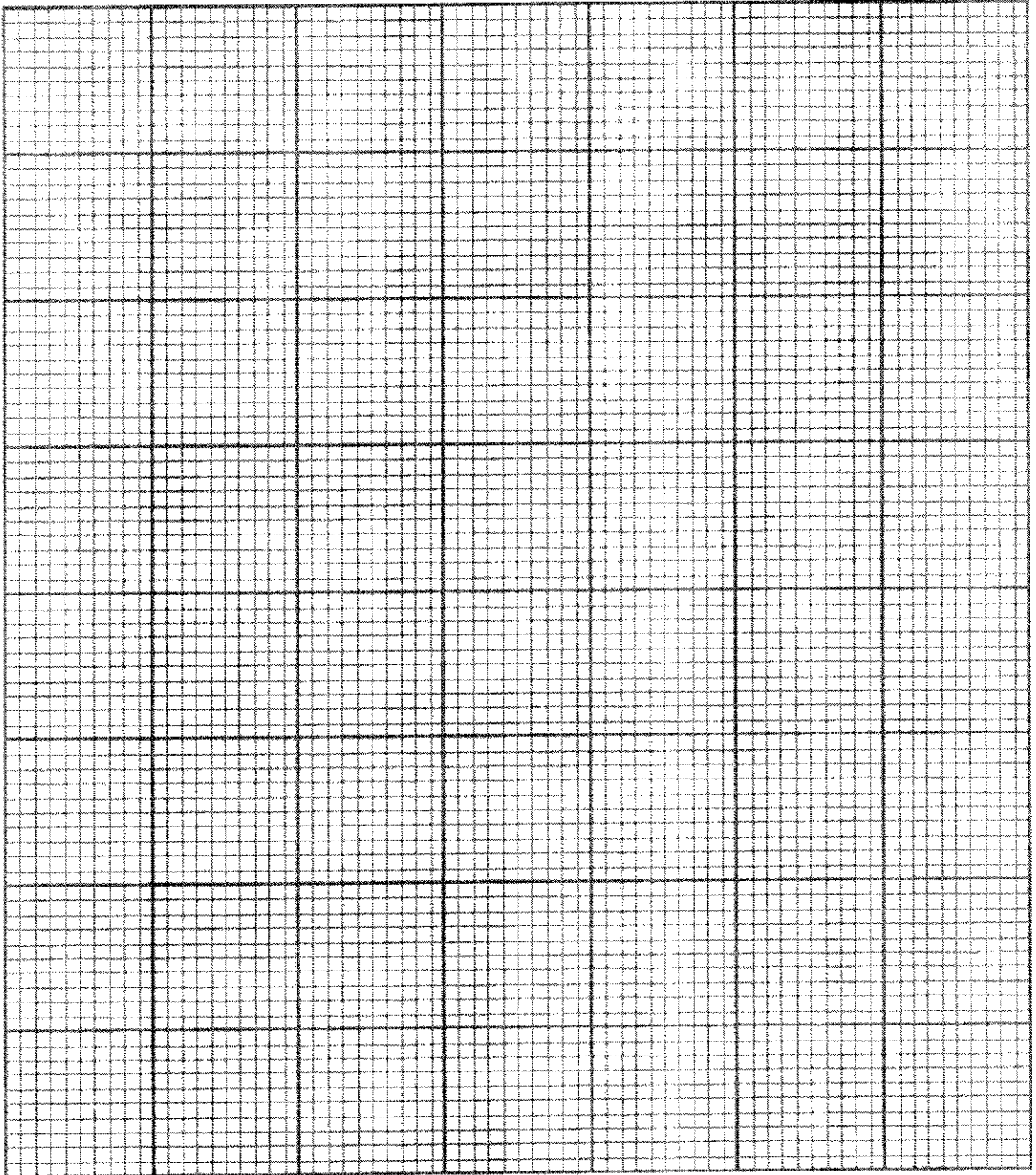| Volume of FA 1 added / cm^3	Temperature / °C
0.00	28.7
5.00	32.7
10.00	35.2
15.00	37.0
20.00	38.2
25.00	39.0
30.00	38.1
35.00	36.6
40.00	35.6
45.00	35.0

[4]

M1   Table has correct headers and units.

M2   Tabulates 10 sets of temp and vol up from 0.00cm^3 to 45.00cm^3
or
Tabulates 9 sets of temp and vol up from 0.50cm^3 to 45.00cm^3, with initial Temp clearly recorded

M3   All volumes are recorded to 2dp, temperatures to nearest 0.1 °C

M4   Temperature increase from 5.00 to 25.00 cm^3 and decreases after 25.00 cm^3 of **FA 1** is added

(b)   Plot a graph of temperature, T, on the y-axis, against total volume of **FA 1** added, on the x-axis. The temperature axis should allow you to include a point at least 2 °C greater than the maximum temperature recorded.

Draw a best-fit straight line taking into account all of the points for the increase in temperature of the mixture. Draw another best-fit straight line taking into account all of the points for the cooling of the solution.

Extrapolate the two lines and determine the
- maximum temperature reached, T_{max},
- maximum temperature increase, ΔT_{max},
- corresponding volume of **FA 1** added.

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T_{max} = ...

ΔT_{max} = ...

Volume of **FA 1** added = ...

[4]

Temperature / °C

T_{max} = 39·7°C

$\Delta T_{max} = 39·7 - 28·7$
$= 11·0°C$

ΔT_{max} = 11·0°C

Volume of **FA1** added = 21·0 cm³

M5 Axes correct way round + correct labels + units + scale (scale must be chosen to allow for the lines to be extrapolated to intersect)
Graph has to occupy more than half the grid for both axes.

M6 Plotting – all points within ±½ small square. (select two from each side to check)

M7 Both graph lines are straight best-fit lines **and** lines are correctly extrapolated to intersect

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M8 T_{max} and volume of FA1 correctly read from the graph to ±½ **small square and** correct ΔT calculated ($T_{initial}$ may come from table or graph plot or intercept on y-axis)

(c)	Explain the shape of the graphs before and after the maximum temperature is reached.
	before maximum temperature is reached
	....
	....
	M9 As more acid was added, temperature rose as the (neutralization reaction between HA and NaOH) **is exothermic / releases heat.**
	after maximum temperature is reached
	....
	....
	M10 NaOH has been **completely neutralized/reacted** and **no more heat is produced / heat is lost to surroundings /heat spread over a larger volume/cooled**
	[2]

(d)	(i)	Calculate the number of moles of HA present in the volume of **FA 1** recorded in **(b).**

		M11 No of moles of HA = 1.80 x (21/1000)
		            = 0.0378 mol
		
		                                  number of moles of HA = ...
		                                                                                              [1]

	(ii)	Using your answers to **(b)**, calculate the heat change, q, when **FA 1** has completely neutralised 25 cm^3 of sodium hydroxide. 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}.

		Total volume = 25 + 21 = 46 cm^3
		
		Q = mcΔT
		   = 46 x 4.18 x 11.0
		   = 2115.08 J
		   = 2120 J
		
		M12 correct total volume
		M13 correct substitution of values into mcΔT (allow ecf)

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		q = ... [2]
	(iii)	Determine the enthalpy change of neutralisation, ΔH_n, for the reaction.
		No of moles of water = no of moles of HA = 0.0378 mol $\Delta H_n = -(q \div \text{moles of water})$ $\qquad = -(2115.08 \times 10^{-3} \div 0.0378)$ $\qquad = -56.0 \text{ kJ mol}^{-1}$ M14 Allow ecf If no negative sign, no marks ΔH_n = ... [1]

	(e)	The maximum error in a thermometer reading is ±0.1 °C. Calculate the maximum percentage error in the increase in temperature recorded.
		M15 Percentage error = $(0.1 \times 2 \div 11) \times 100\% = 1.82\%$ maximum percentage error = ... [1]
	(f)	Apart from using a thermometer with a greater level of precision, suggest one improvement that could be made to the method carried out in (a)
		M16 Use a burette / pipette to measure volume of FA 2 / cover with lid / use smaller volumes close to T_{max} .... [1]
	(g)	A student decided to perform the same experiment in (a) but used aqueous ammonia instead of aqueous sodium hydroxide. Suggest what effect, if any, would replacing aqueous sodium hydroxide with aqueous ammonia have on the value of enthalpy change of neutralisation calculated in d(iii).
		effect .... The value of enthalpy change of neutralisation will be **smaller in magnitude / less exothermic.** (reject larger) explanation .... Aqueous ammonia is a weak base. Some of the **energy released from neutralisation will be absorbed to cause the (complete) dissociation of ammonia**, resulting in the overall heat released to be smaller. M17 [1]
	(h)	Another student repeated the same experiment in (a) but used FB 1 which is 0.9 mol dm^{-3} of HA instead of FA 1.

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		Suggest what effect, if any, would replacing **FA 1** with **FB 1** have on the maximum temperature increase, ΔT_{max}.
		effect .... **M18** ΔT_{max} will be smaller/lowered explanation .... **M19** When [HA] is lowered, a **larger volume of HA will be needed to reach equivalence point**. Since the **same amount of heat is distributed over a larger mass/volume** of solution, the maximum temperature increase will be smaller. [2]

<div align="right">[Total:19]</div>

2	Qualitative analysis

FA 3 contains one cation and two anions
FA 4 contains two cations

Carry out the following tests. Carefully record your observations in **Table 2.1.**

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved.

Table 2.1

		Test	Observations
			FA 3
(a)		Place a spatula of **FA 3** into a test tube, then add approximately 1 cm³ of dilute nitric acid.	No effervescence [√]
			Reject no change/ no observable change/ no reaction
(b)		Place a spatula of **FA 3** into a boiling tube, then add about 15 cm³ of distilled water. Stopper and shake to mix the solution.	
		Use a 1 cm depth of the **FA 3** solution obtained in a test-tube for each of tests **(i) to (iv)**.	
	(i)	Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.	White precipitate [√] soluble in excess. [√]
	(ii)	Add aqueous ammonia slowly, with shaking, until no further change is seen.	White precipitate[√] insoluble in excess.[√]
	(iii)	Add 10 drops of aqueous barium chloride, then add dilute nitric acid, with shaking, until in excess.	White precipitate[√] insoluble in excess. [√]
	(iv)	Add a few drops of aqueous silver nitrate. Decant, then add aqueous ammonia until in excess.	White precipitate [√] soluble in excess ammonia. [√]
			[3]
		2 to 4 [√] – award M20 5 to 7 [√] – award M20 and M21 8 to 9 [√] – award M20, M21 and M22	

(c)	Identify the cation present in **FA 3**. Use evidence from your observations in **Table 2.1** to support your deduction.
	Cation in **FA 3** : ....
	Evidence: ....[1]
	FA 3: Al^{3+} [√] From test b(i) and b(ii) in Table 2.1, **Al^{3+} forms white precipitate with sodium hydroxide, soluble in excess but with ammonia, white precipitate is insoluble in excess.** [√] M23 (allow ecf from b(i) and (ii))
(d)	Identify the anions present in **FA 3**.
	Anion in **FA 3**: .... and ....
	[1]
	Anion in **FA 3**: SO_4^{2-} [√] and Cl^- [√] M24

(e)	FA 4 contains two cations.
	A student added aqueous ammonia to a sample of **FA 4** and observed the formation of a green precipitate that was insoluble in excess of the reagent. He concluded that iron(II) ions were present. Next, he placed a spatula of **FA 4** in a boiling tube and heated the solid gently. The gas evolved turned moist red litmus blue. Upon cooling, a brown residue was obtained.
	(i) Using **Table 2.2**, plan and carry out one other test that will enable you to identify the other cation in **FA 4**. You should start by dissolving half a spatula of **FA 4** in approximately 10 cm³ of distilled water. Use this solution of **FA 4** in your test. Describe your test briefly and state your observations.

Table 2.2

Test	Observations
To 1cm³ of FA 4 solution, **add aqueous sodium hydroxide and warm** [√] the test tube gently.	(Pungent) gas evolved turned moist red litmus blue. [√]

M25
Accept if student did not mention quantity of reagents

			[1]
	(ii)	Identify the other cation in **FA 4** and write an ionic equation for the observation in **e(i)** that allows you to confirm its identity.	
		The other cation in **FA 4**: .... NH_4^+ [√] Ionic Equation: .... $NH_4^+ + OH^- \rightarrow NH_3 + H_2O$ [√] M26	
			[1]
			[Total:7]

3 Determination of the water of crystallisation in hydrated copper(II) sulfate

The addition of an excess of potassium iodide, KI, to a solution of Cu^{2+} ions produces iodine, I_2, and a stable precipitate of CuI. The I_2 turns the solution brown.

equation 1 $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$

The iodine produced can be titrated with a standard solution of $Na_2S_2O_3$ as shown in equation 2.

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

FA 8 is an aqueous solution made by dissolving 32.5 g of $CuSO_4.xH_2O$ in 1.00 dm^3 of solution.

You are also provided with
FA 5, 0.150 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$,
FA 6, dilute sulfuric acid, H_2SO_4,
FA 7, 1.00 mol dm^{-3} potassium iodide, KI,
starch indicator.

In this experiment you will determine the value of x in the formula for hydrated copper(II) sulfate, $CuSO_4.xH_2O$. You will prepare a mixture containing FA 6, FA 7 and FA 8 and titrate the iodine produced against FA 5.

(a)   (i)   Titration of a mixture against FA 5

1. Fill a burette with FA 5.

2. Use a pipette to transfer 25.0 cm^3 of FA 8 into a 250 cm^3 conical flask.

3. Use a measuring cylinder to add 10 cm^3 of FA 6 to the conical flask from step 2.

4. Use a measuring cylinder to add 10 cm^3 of FA 7 to the same conical flask from step 3. A white precipitate forms in a brown solution.

5. Run FA 5 from the burette into the flask. Near the end-point, when the brown solution becomes pale, add about 20 drops of the starch indicator.

6. Continue adding FA 5 slowly. The end-point is reached when the **solution** first becomes colourless. The white precipitate remains.

7. Record your titration results, to an appropriate level of precision, in the space provided on Page 12.

8. Discard the contents of the conical flask **immediately** down the sink. Wash out the conical flask thoroughly with tap water.

9. Repeat points 2 to 8 until consistent results are obtained.

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Titration results

Accuracy : Supervisor's average titre = 21.67 cm^3

[6]

final burette **reading**/ cm^3	22.60	22.60
initial burette **reading**/ cm^3	0.00	0.00
volume of **FA 5** added/ cm^3	**22.60**	**22.60**

M27 Tabulates initial and final burette readings and volumes added in the titration table. Table has correct headers and units. *Tabulation may be vertical or horizontal; lines are not essential but there should be no absences of headers. Where units have not been included in the header, there should be the appropriate unit for each entry in the table.*
DO NOT award this mark if any final and initial burette readings are inverted or 50 is used as the initial burette reading or any burette reading is greater than 50.

M28 All the final/initial burette readings, for all accurate titres in the titration table, are recorded to the nearest 0.05 cm^3 and readings entered correctly that is final reading is greater than initial reading. *Treat all titres as "accurate" unless labelled "rough" or first titre is recorded to lower precision than subsequent titres*

M29 Has at least two <u>uncorrected</u> titres for end-point within 0.10 cm^3. (ignore calculation error, we will just take student's value at face value)

M30 to M32 Calculate the student's mean titre as described:
Hierarchy to be used in calculating mean titres in question 3(a)(ii):
Value of 2 identical titres
Average of titres within 0.05 cm^3
Average of titres within 0.1 cm^3, etc.
Award based on the difference, $\Delta titre$, between Student's and Supervisor's mean titre.
Award M30, M31 and M32 if this difference is $\leq$ **0.20 cm^3**
Award M30 and M31 marks if this difference is > **0.20 cm^3** but $\leq$ **0.40 cm^3**
Award M30 mark if this difference is > **0.40 cm^3** but $\leq$ **0.60 cm^3**
Award 0 mark for a difference > **0.60 cm^3**

(ii) From your titrations, obtain a suitable volume of **FA 5** to be used in your calculations. Show clearly how you obtained this volume.

Volume of **FA 5** = ...[1]

V_{FA5} = (22.60 + 22.60) ÷ 2 = **22.60 cm^3** [1]

M33 Student calculates the average correctly to 2dp, from any experiments with uncorrected end point titre values within 0.10 cm^3. The titre values used must be shown in calculation **or** ticks must be put next to the two accurate readings selected.
DO NOT award this mark if the titres used are not identified or if there are arithmetic errors in the table.

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(b) **(i)** Calculate the amount of copper(II) ions, Cu^{2+}, in 25.0cm³ of **FA 8**.

$$\text{no of moles of } S_2O_3{}^{2-} = 0.150 \times \frac{\textbf{22.60}}{1000} = 0.00339 \text{ mol}$$

M34   no of moles of Cu^{2+} = no of moles of $S_2O_3{}^{2-}$ = 0.00339 mol

amount of Cu^{2+} in **FA 8** = ...[1]

(ii) Calculate the concentration of copper(II) ions, $[Cu^{2+}]$, of **FA 8**.

$$\textbf{M35 } \text{concentration of } Cu^{2+} = \frac{\textbf{0.00339}}{0.0250} = 0.1356 = 0.136 \text{ mol dm}^{-3}$$

Allow ecf

concentration of Cu^{2+} in **FA 8** = ... [1]

(iii) Use your answer from **(b)(ii)** to calculate the M_r of hydrated copper(II) sulfate.

$$\textbf{M36 } M_r \text{ of } CuSO_4.xH_2O = \frac{32.5}{\textbf{0.1356}} = 239.7$$

Allow ecf

M_r of hydrated copper(II) sulfate = ...

Hence, deduce the value of **x**.
Show your working.
[A_r: H, 1.0; O, 16.0; S, 32.1; Cu, 63.5]

$$\textbf{M37 } \textbf{x} = \text{nearest integer to } \frac{239.7 - 159.6}{18.0} = 4.45$$
$$= 4 \quad \text{(need to be whole number)}$$

Allow ecf

x = ... [5]

M38 – working: Shows workings in all calculations in **1d(i), (ii), (iii), 1(e), 3b(i), (ii), (iii)** All calculations must be relevant (must include some data in the calculation, merely writing formula has no marks) although they may not be complete or correct. Any calculation not attempted loses this mark.

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M39 – s.f.: Shows appropriate significant figures in all final answers in **1d(i), (ii), (iii),** **1(e), 3b(i), (ii), (iii).** Any calculation not attempted loses this mark.

M40 – units: Shows appropriate units in all final answers in **1b, 1d(i), (ii), (iii), 3a(ii),** **3b(i), (ii), (iii).** Any calculation not attempted loses this mark.

(c) Identify two different chemical processes that use iodide ions in this experiment.

....

....

[1]

M41 Redox and precipitation.

(d) A student suggests that the experiment could be made more accurate if the volume of **FA 7** was measured using a burette.

(i) Give a reason why the student might make this suggestion.

....

....

[1]

M42 The burette has higher precision or smaller uncertainty or percentage error is smaller. Reject higher accuracy.

(ii) Explain whether this change will improve the accuracy of the experiment.

....

....

[1]

M43 Using a burette to measure the volume of KI will **not** improve the accuracy as KI is in **excess**.

[Total: 17]

4	Planning

The method of initial rates is a commonly used technique for deriving rate laws. To determine the initial rate of a reaction, we can time how long it takes to reach an identifiable point early in the reaction.

In solution, iodide ions, I^-, are oxidised by peroxodisulfate ions, $S_2O_8^{2-}$.

$$S_2O_8^{2-} \text{ (aq)} + 2I^- \text{ (aq)} \rightarrow 2SO_4^{2-} \text{ (aq)} + I_2 \text{ (aq)}$$

If sodium thiosulfate and starch are added to the reaction mixture, the blue-black colour of an iodine-starch complex appears suddenly after some time. This occurs when all of the thiosulfate ions, $S_2O_3^{2-}$, present in the mixture have reacted with the iodine formed in the reaction above. This is the identifiable point in the reaction.

$$2S_2O_3^{2-} \text{ (aq)} + I_2 \text{ (aq)} \rightarrow S_4O_6^{2-} \text{ (aq)} + 2I^- \text{ (aq)}$$

You are to plan an experiment to investigate how the rate of reaction between potassium peroxodisulfate and potassium iodide depends on the concentration of potassium peroxodisulfate based on a graphical analysis.

A preliminary experiment, using approximate volumes of solution, indicates that the time taken for the iodine-starch complex to form doubles when the potassium peroxodisulfate is diluted with an equal amount of water.

	(a)	Using the results of the preliminary experiment, predict the order of reaction with respect to potassium peroxodisulfate. Explain your answer.

....

....

[2]

M44 First order [1]
M45 because when the **[potassium peroxodisulfate] is halved**, the **rate is halved** [1] or the rate of reaction is **directly proportional** to [potassium peroxodisulfate].

	(b)	Explain why it is important that the iodine formed by oxidation reacts with the sodium thiosulfate and is converted back to iodide ions.

....

....

....

[1]

M46 [iodide] needs to remain constant / [iodide] is a controlled variable, so it's important to form the iodide ions so that there's continuous supply of iodide ions, so that (rate w.r.t. peroxodisulfate can be studied)

	(c)	Explain why the volume of aqueous sodium thiosulfate should be measured precisely, using a burette, in the experiment.

....

....

[1]

M47 So that we can investigate the time taken for an <u>**exact and fixed amount of iodine to be formed**</u>.

Accept also: This is because <u>**volume / amount of thiosulfate ions define the extent of the reaction**</u> ('finishing line'). To <u>**enable fair comparison**</u>, the constant volume of thiosulfate to the end point must be measured precisely.

<u>Reject: smaller percentage error if use burette</u>

(d)		Consider the description of the experiment given in the previous page.

Write a plan for a series of experiments to investigate your prediction in **(a)**.

You may assume that you are provided with:

- 0.60 mol dm^{-3} potassium iodide
- 0.20 mol dm^{-3} potassium peroxodisulfate
- 0.01 mol dm^{-3} sodium thiosulfate
- distilled water
- starch indicator solution
- the equipment normally found in a school laboratory

A first experiment is carried out using the following quantities.

- 20 cm^3 potassium iodide
- 40 cm^3 potassium peroxodisulfate
- 20 cm^3 sodium thiosulfate
- 0 cm^3 distilled water
- 10 cm^3 starch indicator solution

Give a step-by-step description of the method you would use in further experiments.

Your plan should contain the following:

- the apparatus you would use,
- a table to indicate the different quantities of reagents you would use,
- the procedure you would follow,
- the measurements you would make to allow a suitable graph to be drawn in order to investigate your prediction in **(a)**.

[6]

....

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Proposed Answer:

Expt no.	Volume of potassium peroxodisulfate / cm^3	Volume of KI/ cm^3	Volume of sodium thiosulfate / cm^3	Volume of water/ cm^3	Volume of starch indicator solution / cm^3
1	40	20	20	0	10
2	30	20	20	10	10
3	20	20	20	20	10
4	10	20	20	30	10
5	5	20	20	35	10

Procedures:

1	✓ Use different dry **50cm³ measuring cylinders**, measure 20 cm^3 of KI and 10 cm^3 of starch solution separately into a **250 cm^3 conical flask/beaker**. [remember: this mixture should not react with each other]
2	✓ Using a burette, measure 20.00 cm^3 of $Na_2S_2O_3$ into the same conical flask/beaker.
3	✓ Use another different dry 50 cm^3 measuring cylinder, add 40 cm^3 of aqueous potassium peroxodisulfate into the same conical flask/beaker.
4	✓ Start the stopwatch immediately. Swirl the conical flask/beaker.

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		5	✓ Stop the stopwatch and record the time when the solution turns blue-black.	
		6	✓ Repeat steps 1 to 5 and vary the volume of potassium peroxodisulfate and water according to the table above.	
		/		

Mark Scheme

		Remarks
M48	A table indicating at least 3 more appropriate experiments of lower concentration/volumes of peroxodisulfate (inappropriate experiments negate this mark) + at least one of the diluted solutions is half or less of original given concentration/ uses 20 cm³ or less of peroxodisulfate at least once.	-
M49	Total volume, volumes of KI, $S_2O_3^{2-}$, and starch all constant	
M50	Appropriate apparatus, with capacity stated M3 can only be awarded for complete plan (can be wrong/right)	Burette for measuring $Na_2S_2O_3$ not marked for since it is given in (c)
M51	Procedure to prepare reaction mixtures and start the mixing: Steps 1 to 3 (peroxodisulfate and iodide kept apart until reaction stated has started)	
M52	Procedure to record time + describes a sensible 'end-point'; time needed for the reaction mixture to turn dark blue: Steps 4 to 5	
M53	Mentions about repeating the procedure: Step 6	

(e) Assuming that your prediction in **(a)** is correct, sketch on Fig. 4.1 the graph you would expect to obtain from your results. Label the axes.

rate or 1/t

Fig. 4.1

Volume of potassium peroxodisulfate

M54 – axes labels
M55 – shape of graph (Allow ecf based on answer in (a))
Reject concentration-time graph

[2]

[Total: 12]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	reaction with	
	NaOH(aq)	NH₃(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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

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(b) Reactions of anions

ions	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 → (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) Test for gases

ions	reaction
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

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