



TAMPINES MERIDIAN JUNIOR COLLEGE

JC2 PRELIMINARY EXAMINATION

CANDIDATE NAME **SUGGESTED SOLUTIONS**

CIVICS GROUP 21S

H2 CHEMISTRY

Paper 4 Practical

9729

30 August 2022

2 hours 30 minutes

Candidates answer on the Question Paper.

READ THESE INSTRUCTIONS FIRST

Write your name and Civics Group in the spaces at the top of the 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 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.

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

Shift
1 / 2 / 3
Laboratory

For Examiner's Use	
1	/ 11
2	/ 20
3	/ 10
4	/ 14
Total	/ 55

This document consists of **22** printed pages.

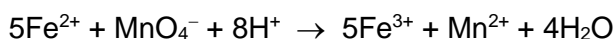


1 Determination of water of crystallisation in a hydrated iron(III) salt

A variety of hydrated iron(III) sulfates are known. Solutions of iron(III) sulfate are used in dyeing, and as coagulant for industrial waste.

FA 1 is a solution containing 26.0 g dm^{-3} of hydrated iron(III) sulfate, $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$. The addition of excess zinc to a solution of **FA 1** reduces the Fe^{3+} ions to Fe^{2+} ions.

The amount of Fe^{2+} ions can be determined quantitatively by titration against a standard solution of potassium manganate(VII), KMnO_4 . The reaction is shown below.



In this experiment, you are to perform titrations to determine the value of n , the water of crystallisation in **FA 1**.

You are provided with

FA 1, solution containing 26.0 g dm^{-3} of hydrated iron(III) sulfate, $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$.

FA 2, dilute sulfuric acid,

FA 3, $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 ,
zinc powder

(a) Preparation of Fe^{2+} solution from **FA 1**

1. Use a measuring cylinder to transfer 80 cm^3 of **FA 1** into a 250 cm^3 beaker.
2. Add cautiously all the zinc powder into the beaker. Cover the beaker with a white tile.
3. Allow the reaction to take place for about 5 minutes, stirring the reaction mixture from time to time.
4. Filter the mixture into the **dry** beaker provided using a **dry** filter paper and filter funnel. Ignore any reaction that may still be taking place.
5. Label the filtrate as **FA 4**. Proceed to **1(b)** once you have collected sufficient filtrate.

(b) Titration of **FA 4** against **FA 3**

6. Fill the burette labelled **FA 3** with **FA 3**.
7. Use a pipette to transfer 10.0 cm^3 of **FA 4** into a 100 cm^3 conical flask.
8. Use a measuring cylinder to add 10 cm^3 of **FA 2** to this flask.
9. Titrate **FA 4** with **FA 3** from the burette until the appearance of the first permanent pale pink colour.
10. Record your titration results, to an appropriate level of precision, in the space provided on page 3.
11. Repeat steps 7 to 10 until consistent results are obtained.

Wash out the conical flasks and stand it upside down to drain for use in **Question 2**.



(i) Results

Final burette reading / cm ³	9.90	19.80
Initial burette reading / cm ³	0.00	9.90
Volume of FA 3 used / cm ³	9.90	9.90
	✓	✓

[3]

- (ii) From your titrations, obtain a suitable volume of **FA 3**, $V_{\text{FA 3}}$, to be used in your calculations. Show clearly how you obtained this volume.

$$\begin{aligned}\text{Average volume of FA 3} &= (9.90 + 9.90) \div 2 \\ &= \underline{9.90 \text{ cm}^3}\end{aligned}$$

$$V_{\text{FA 3}} = \dots\dots\dots [3]$$

M1	M2	M3	M4	M5	M6

- (c) (i) Calculate the amount of Fe^{2+} in 10.0 cm³ of **FA 4**.



$$\begin{aligned}\text{Amount of Fe}^{2+} \text{ in } 10.0 \text{ cm}^3 \text{ of FA 4} &= \frac{9.90}{1000} \times 0.0200 \times 5 \\ &= 9.90 \times 10^{-4} \text{ mol (3 s.f.)}\end{aligned}$$

$$\text{amount of Fe}^{2+} \text{ in } 10.0 \text{ cm}^3 \text{ of FA 4} = \dots\dots\dots \text{ mol [1]}$$

M7	
----	--

- (ii) In step 2, an excess of zinc was added to convert the Fe^{3+} to Fe^{2+} .

Calculate the amount of Fe^{3+} in 1 dm³ of **FA 1**.

$$\begin{aligned}\text{Amount of Fe}^{3+} \text{ in } 1 \text{ dm}^3 \text{ of FA 1} &= 9.90 \times 10^{-4} \times \frac{1000}{10.0} \\ &= 9.90 \times 10^{-2} \text{ mol (3 s.f.)}\end{aligned}$$

$$\text{amount of Fe}^{3+} \text{ in } 1 \text{ dm}^3 \text{ of FA 1} = \dots\dots\dots \text{ mol [1]}$$

M8	
----	--



- (iii) Use your answer from (c)(ii) to calculate the M_r of the hydrated iron(III) sulfate, $\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$, in **FA 1**.



$$\begin{aligned} \text{Amount of } \text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O} \text{ in } 1 \text{ dm}^3 \text{ of FA 1} &= \frac{1}{2} \times 9.90 \times 10^{-2} \text{ mol} \\ &= 4.95 \times 10^{-2} \text{ mol (3 s.f.)} \end{aligned}$$

$$M_r \text{ of } \text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O} = \frac{26.0}{4.95 \times 10^{-2}} = 525.3 \text{ (no units)}$$

M_r of the hydrated iron(III) sulfate =

Hence, deduce the value of n , the water of crystallisation in the hydrated iron(III) sulfate.

[A_r : Fe, 55.8; S, 32.1; O, 16.0; H, 1.0]

$$\begin{aligned} n &= \{525.3 - [2(55.8) + 3(32.1) + 12(16.0)]\} \div 18.0 \\ &= 125.4 \div 18.0 \\ &= 6.96 \\ &= 7 \text{ (nearest whole number)} \end{aligned}$$

$n =$ [2]

M9	M10

- (d) In step 4, excess zinc was filtered off before titration of **FA 4** against **FA 3**.

Suggest why it was necessary to filter off the excess zinc metal, and what effect it would have on the titre values if this filtration was not carried out.

Any zinc metal that is not removed will reduce Fe^{3+} formed during the titration to Fe^{2+} , resulting in a higher than expected titre / volume of FA3 (KMnO_4) used.

..... [1]

M11	
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[Total: 11]



2 Determination of the kinetics of the reaction between M^{3+} ions and iodide ions, I^- .

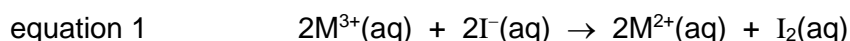
You are provided with the following reagents.

FA 5 contains $0.0200 \text{ mol dm}^{-3}$ metal ions, M^{3+} , also present in **FA 9**.

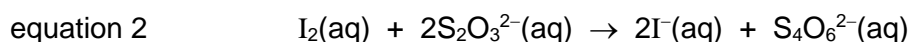
FA 6 is $0.0080 \text{ mol dm}^{-3}$ aqueous potassium iodide, KI

FA 7 is $0.0060 \text{ mol dm}^{-3}$ sodium thiosulfate, $Na_2S_2O_3$
starch solution

M^{3+} ions oxidise iodide ions, I^- , to iodine, I_2 as shown in equation 1. In this experiment, you will investigate how the rate of this reaction is affected by the concentration of M^{3+} ions.



A fixed and small amount of thiosulfate ions, $S_2O_3^{2-}$, and starch indicator will be added to a mixture of $M^{3+}(aq)$ and $I^-(aq)$. The iodine, I_2 , produced reacts immediately with thiosulfate ions, $S_2O_3^{2-}$ as shown in equation 2.



When all the thiosulfate has been used, the iodine produced will turn starch indicator blue-black. The rate of the reaction can therefore be measured by finding the time it takes for the reaction mixture to turn blue-black.

You will perform a series of **four** experiments. Then, you will graphically analyse your results to determine the order with respect to the concentration of M^{3+} ions, $[M^{3+}]$.

For each experiment, you will note the volume of **FA 5** added, $V_{FA\ 5}$, and the time taken, t , for the reaction mixture to become blue-black. In each experiment, you will need to ensure that the same total volume of reaction mixture is used by adding deionised water as required.

You will then calculate values for

- $\frac{1}{t}$,
- $\lg\left(\frac{1}{t}\right)$,
- $\lg(V_{FA\ 5})$.

(a) Prepare a table in the space provided on page 7 in which to record, to an appropriate level of precision:

- volumes of **FA 5** and deionised water,
- all values of t ,
- all calculated values of $\frac{1}{t}$, $\lg\left(\frac{1}{t}\right)$ and $\lg(V_{FA\ 5})$.



(i) Experiment 1

1. Fill a burette with **FA 5**.
2. Transfer 20.00 cm³ of **FA 5** into a 100 cm³ conical flask.
3. Use the measuring cylinders to place the following in a 100 cm³ beaker.
 - 10 cm³ of **FA 6**
 - 15 cm³ of **FA 7**
 - 10 cm³ of starch solution
4. Add the contents of the beaker rapidly to the conical flask and start the stopwatch.
5. Swirl the mixture and place the conical flask on the white tile.
6. The mixture turns purple and then yellow before turning a blue-black colour. Stop timing when this **blue-black colour first** appears.
7. Record the time taken, t , to nearest second in your table.
8. Discard the reaction mixture **immediately** down the sink. Wash out the conical flask and stand it upside down on a paper towel to drain.

(ii) Experiment 2

1. Run 8.00 cm³ of **FA 5** into a 100 cm³ conical flask.
2. Using another measuring cylinder, add 12.0 cm³ of deionised water into the conical flask containing **FA5**.
3. Use the measuring cylinders from Experiment 1 to place the following in a 100 cm³ beaker.
 - 10 cm³ of **FA 6**
 - 15 cm³ of **FA 7**
 - 10 cm³ of starch solution
4. Add the contents of the beaker rapidly to the conical flask and start the stopwatch.
5. Swirl the mixture and place the conical flask on the white tile.
6. The mixture turns purple and then yellow before turning a blue-black colour. Stop timing when this **blue-black colour first** appears.
7. Record the time taken, t , to nearest second in your table.
8. Discard the reaction mixture **immediately** down the sink. Wash out the conical flask and stand it upside down on a paper towel to drain.

In Experiment 1 you will have obtained the time taken for a 'fast' reaction and in Experiment 2 the time taken for a 'slow' reaction.

Carry out **two** further experiments to investigate the effect of changing the concentration of M³⁺(aq) by altering the volume of M³⁺, **FA 5**, used. In each case, you will need to ensure that the **same total volume** of reaction mixture is used by adding deionised water as required.

Do not use a volume of **FA 5** that is less than 8.00 cm³.

You should alternate the use of the two 100 cm³ conical flasks.

Record all required volumes, time taken and calculated values in your table.



(iii) Results

Expt	Volume of FA 5 / cm ³	Volume of deionised H ₂ O / cm ³	time / s	$\frac{1}{t}$ / s ⁻¹	lg (V _{FA 5})	lg $\left(\frac{1}{t}\right)$
1	20.00	0.0	16	0.0625	1.30	-1.20
2	8.00	12.0	37	0.0270	0.903	-1.57
3	12.00	8.0	25	0.0400	1.08	-1.40
4	16.00	4.0	19	0.0526	1.20	-1.28

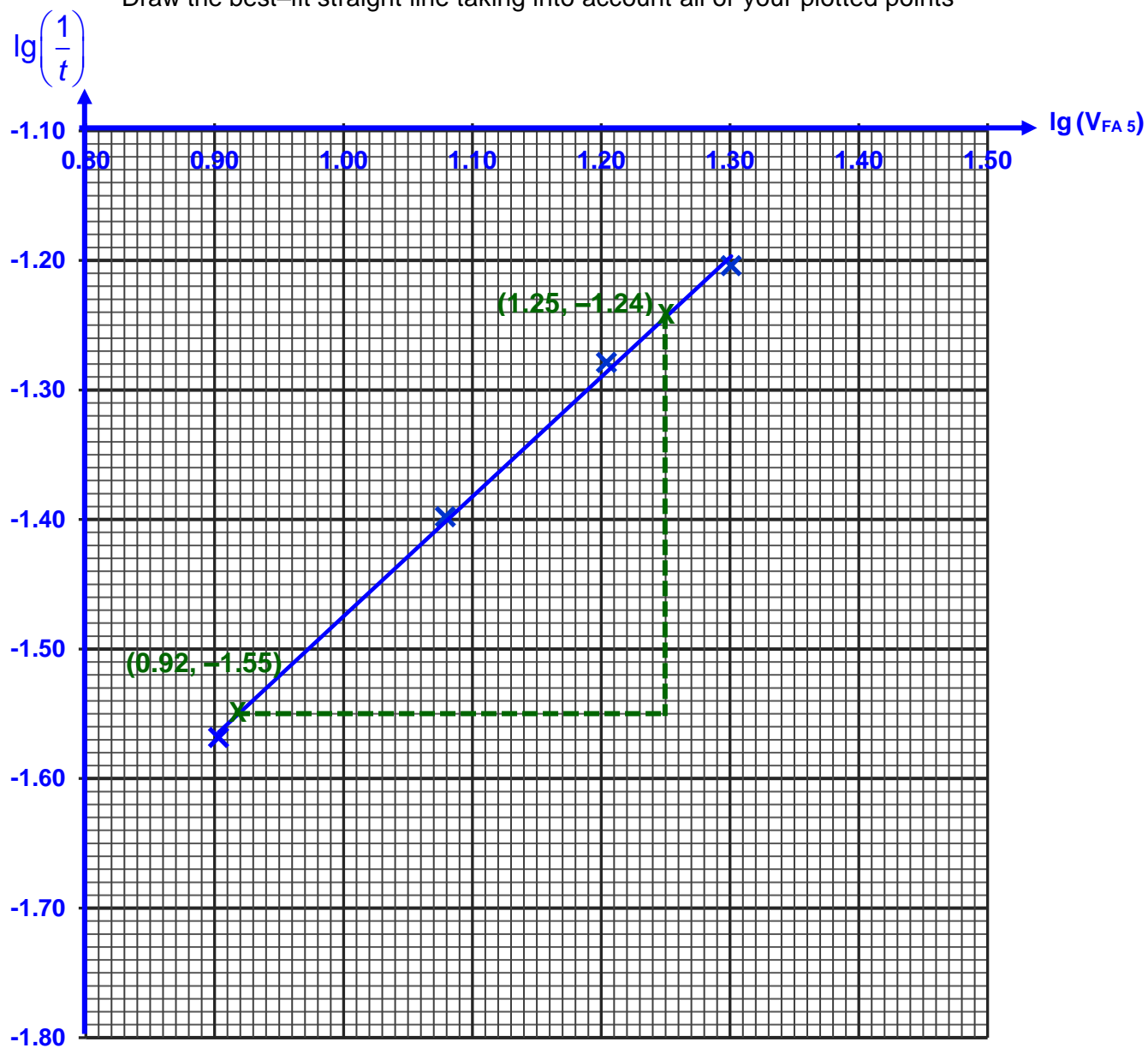
[5]

M12	M13	M14	M15	M16



- b) (i) Plot a graph of $\lg\left(\frac{1}{t}\right)$ on the y-axis against $\lg(V_{FA5})$ on the x-axis.

Draw the best-fit straight line taking into account all of your plotted points



[3]

M17	M18	M19

- (ii) Calculate the gradient of the line to three significant figures, showing clearly how you did this. Hence, deduce the order of the reaction with respect to $[M^{3+}]$.

$$\text{Gradient} = \frac{-1.24 - (-1.55)}{1.25 - 0.92} = \mathbf{0.939} \text{ (3 s.f.)}$$

Hence, the **gradient of the graph of $\lg\left(\frac{1}{t}\right)$ vs $\lg(V_{FA})$** is the order of the reaction w.r.t. $[M^{3+}]$. **Order of reaction with respect to $[M^{3+}]$ is 1.**

gradient =

order =

[3]

M20	M21	M22

- (c) When you performed this experiment, you were instructed to wash **and** drain a conical flask before using it again.

State and explain the likely effect on t of **not** draining a flask before it is reused.

effect on t The time taken will be **longer than expected**.

explanation The **residual water present** in the conical flask **decreases the concentration of the reactants** in the reaction mixture, **decreasing the rate of reaction**.

[1]

M23	
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- (d) Explain why a small and fixed amount of sodium thiosulfate was added in each reaction mixture.

As the iodine is produced, it reacts immediately with the thiosulfate ions. When all thiosulfate has reacted, the iodine then turns the starch indicator blue-black. The **initial rate of reaction** may be determined by measuring the time taken for the reaction mixture to turn blue-black which is equivalent to **the time taken for the same small amount of iodine to be produced (amount needed to react with small and fixed amount of thiosulfate completely)**.

M24	
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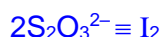


- (e) The rate of this reaction relative to $[M^{3+}]$ can be determined using the following expression. The change in the concentration of $M^{3+}(aq)$ can be determined at the point when sufficient iodine was produced for the appearance of the blue-black colour.

$$rate = \left| \frac{\Delta [M^{3+}]}{\Delta t} \right|$$

- (i) Calculate the amount of iodine that reacted with the thiosulfate ions used in each experiment in (a).

$$\text{Amount of thiosulfate ions} = \frac{15.0}{1000} \times 0.0060 = 9.000 \times 10^{-5} \text{ mol}$$



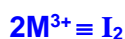
$$\text{Amount of iodine that reacted} = (9.00 \times 10^{-5}) \div 2 = \underline{4.50 \times 10^{-5} \text{ mol}}$$

amount of I_2 = [1]

M25	
-----	--

- (ii) Calculate the amount of M^{3+} ions that was required to produce the amount of iodine in (e)(i). Hence, calculate the rate of this reaction relative to $[M^{3+}]$ for **Experiment 1** in **2(a)** when the blue-black colour first appears.

The change in concentration of M^{3+} ions is due to a change in the amount of M^{3+} ions that reacted with iodide ions to produce the blue-black colour.



$$\begin{aligned} \text{Amount of } M^{3+} \text{ ions required to produce } 4.50 \times 10^{-5} \text{ mol of } I_2(aq) \\ &= (4.50 \times 10^{-5}) \times 2 \\ &= \underline{9.00 \times 10^{-5} \text{ mol (3 s.f.)}} \end{aligned}$$

$$\begin{aligned} \text{Change in } [M^{3+}] &= (9.000 \times 10^{-5}) \div \frac{55.0}{1000} \text{ (Total volume of reaction mixture)} \\ &= 1.636 \times 10^{-3} \text{ mol dm}^{-3} = 1.64 \times 10^{-3} \text{ mol dm}^{-3} \end{aligned}$$

$$rate = \left| \frac{\Delta [M^{3+}]}{\Delta t} \right| = \left| \frac{1.636 \times 10^{-3}}{\text{time taken in Expt 1}} \right| = \left| \frac{1.636 \times 10^{-3}}{16.0} \right| = \underline{1.02 \times 10^{-4} \text{ mol dm}^{-3} \text{ s}^{-1}}$$

(3 s.f.)

Amount of M^{3+} ions required =

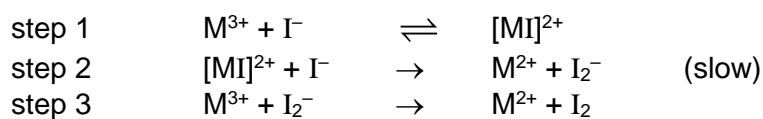
rate of reaction = mol dm⁻³ s⁻¹

[4]

M26	M27	M28	M29



- (f) The following steps represent a possible mechanism for the reaction shown in equation 1



State which step is the rate-determining step. Hence, derive the rate law for this reaction.

Step 2 is the rate determining step.

Rate = $k [MI]^{2+} [I^-]$

Since $[MI]^{2+}$ is an intermediate formed from M^{3+} and I^- in the preceding fast step,

$[MI]^{2+} \propto [I^-] [M^{3+}] \rightarrow \text{Rate} = k' [M^{3+}] [I^-]^2$ [2]

M30	M31

[Total: 20]



3 Planning

A student suggested that the temperature at which Experiment 1 in **2(a)(i)** was carried out will also affect the rate of the reaction.

The activation energy, E_a , and the pre-exponential factor, A , which is a constant, can be determined from the equation.

$$k' = Ae^{\frac{-E_a}{RT}}$$

T is the reaction temperature in Kelvin.

k' is the rate constant at a chosen temperature.

The procedure you followed for Experiment 1 in **2(a)(i)** can be modified and extended to investigate the effect of temperature, T , on the rate of the reaction between M^{3+} and I^- . The activation energy, E_a , and the pre-exponential factor, A , can be graphically determined.

Plotting $\ln k'$ against $\frac{1}{T}$ gives a straight line of best fit. The gradient of this line is $\frac{-E_a}{R}$, where R is the molar gas constant.

- (a) Plan an investigation, based on Experiment 1 described in **2(a)(i)**, to determine the effect of temperature on the rate of reaction.

You may assume that you are provided with the same reagents as experiment in **2(a)(i)** as well as the equipment normally found in a school laboratory

In your plan, you should include brief details of

- the reactants and conditions that you would use,
- the apparatus that you would use **in addition** to that specified in Experiment 1 of **2(a)(i)**,
- the modification/ extension of procedure required in addition to the procedures spelt out in Experiment 1 of **2(a)(i)**
- the measurements that you would take and how you would determine the rate for each experiment.

FA 5 contains $0.0500 \text{ mol dm}^{-3}$ metal ions, M^{3+}

FA 6 is $0.0080 \text{ mol dm}^{-3}$ aqueous potassium iodide, KI

FA 7 is $0.0060 \text{ mol dm}^{-3}$ sodium thiosulfate, $Na_2S_2O_3$

starch solution

1. Fill a burette with **FA 5**.
2. Transfer 20.00 cm^3 of **FA 5** into a 100 cm^3 conical flask.
3. Use the measuring cylinders to place 10 cm^3 of **FA 6**, 15 cm^3 of **FA 7** and 10 cm^3 of starch solution in a 100 cm^3 beaker.
4. Place the conical flask and beaker in a thermostatically controlled water-bath, set at 30°C .



5. After a few minutes, measure and record the temperature of contents in the beaker and/ or the conical flask using a thermometer.
6. Add the contents of the beaker rapidly to the conical flask, while keeping the conical flask in the thermostatically controlled water-bath. Start the stopwatch and swirl the mixture.
7. Stop timing when this blue-black colour first appears. Record the time taken, t , to 0.1 s in your table.
8. Discard the reaction mixture immediately down the sink. Wash out the conical flask and stand it upside down on a paper towel to drain.
9. Repeat steps 2 to 9 at three other temperatures at 40°C, 50°C and 60°C by placing the conical flask in a thermostatically controlled water-bath over the course of the reaction.
10. Calculate the rate of reaction by taking the reciprocal of the time taken, t , for the reaction mixture to become blue-black, rate = $1/t$.

[4]

M32	M33	M34	M35



- (b) Briefly describe how you would use results obtained from 3(a) to determine all necessary values in order to plot a graph of $\ln k'$ against $\frac{1}{T}$.

You do not need to perform any of the calculations.

To obtain $\ln k'$:

Using the **rate of experiments from 3(a)**, the value of k' can be found using the **rate equation, $\text{Rate} = k' [\text{reactants}]^m$** , and calculating $\ln k'$.

To obtain $1/T$:

Convert temperature recorded in $^{\circ}\text{C}$ to K, followed by taking reciprocal to find $1/T$.

[2]

M36	M37

- (c) Sketch the graph you would expect to obtain from 3(b) on the axes in Fig. 3.1. Explain your answer.

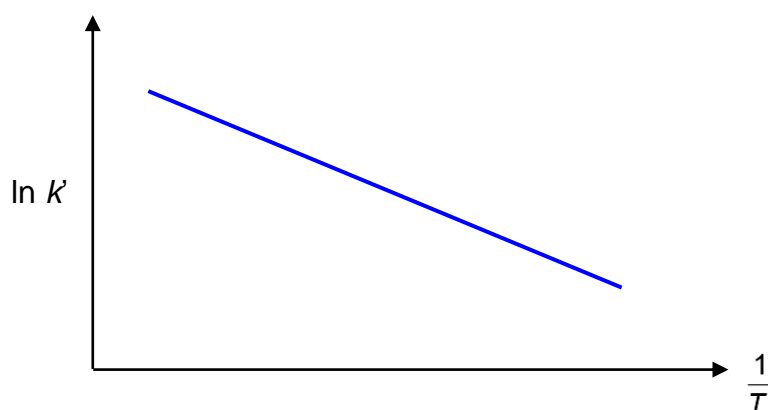


Fig. 3.1

explanation When temperature (or T) increases, rate of reaction increases.

When T is large, $\frac{1}{T}$ will be a small value.

When rate of reaction increases, value of $\ln k'$ will also increase.

Overall, when $\frac{1}{T}$ increase, $\ln k'$ will decrease.

[2]

OR

Since E_a is positive, and R is positive,
gradient of graph = $-E_a / R$ must be a negative value.

M38	M39

- (d) Describe how you would use your graph in (c) to determine values for E_a and A .

E_a

Determine the gradient of the graph of $\ln k$ against $1/T$. The value of the gradient is equivalent to the value of (E_a / R) .

To determine E_a ; $E_a = \text{Gradient of graph} \times R$ (Molar gas constant)

E_a will have unit of J mol^{-1} .

A

Extrapolate the graph to determine the y-intercept of the graph of $\ln k$ against $1/T$. The value of the y-intercept of the graph is equivalent to the value of $\ln A$.

To determine the value of A , $A = e^{\text{value of the y-intercept}}$

M40	M41

[Total: 10]



4 Investigation of some inorganic and organic reactions

In this question, you will be investigating some inorganic and organic reactions by carrying out the stipulated tests. Carefully record your observations in **Tables 4.1** and **4.2**.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

(a) Organic analysis

In this question, you will deduce the structure of an organic compound, **FA 8**. **FA 8** has the molecular formula $C_4H_8O_2$ with **two** functional groups present.

Do not carry out the tests for which observations have been recorded.

Do not use the Bunsen burner for heating in this part of the question. Instead, use the hot water provided.

Table 4.1

	tests	observations
(i)	Place about 2 cm depth of aqueous sulfuric acid in a test-tube. To this test-tube, add about 1 cm depth of FA 8 , followed by 1 drop of aqueous potassium manganate(VII). Warm the mixture in the hot water bath for two minutes.	<ul style="list-style-type: none"> • Purple decolourises. $KMnO_4$
(ii)	Add 1 cm depth of aqueous silver nitrate to a test-tube. Then slowly add 1 cm depth of aqueous sodium hydroxide. Add aqueous ammonia slowly, with shaking, until the precipitate just dissolves. To this mixture, add about 1 cm depth of FA 8 . Place the test-tube containing the mixture in the water bath for one minute.	<ul style="list-style-type: none"> • Brown / grey ppt formed • Brown / grey ppt dissolves to give a colourless solution • No silver mirror observed / No observable change
(iii)	Place about 1 cm depth of FA 8 in a test-tube. To this test-tube, add 2,4-dinitrophenylhydrazine dropwise.	Orange ppt. formed

	tests	observations
(iv)	Place about 1 cm depth of FA 8 and add 8 drops of aqueous sodium hydroxide in a test-tube. Now add iodine solution dropwise, until a permanent yellow / orange colour is obtained. Warm the mixture in the hot water bath for two minutes.	<ul style="list-style-type: none"> • Pale yellow ppt (in yellow / orange solution) observed.
(v)	Place 1 cm depth of FA 8 in a test-tube. To this test-tube, cautiously add a small piece of sodium metal.	Effervescence observed H ₂ gas produced extinguishes a lighted splint with a "pop" sound.

[2]

M42	M43

- (b) (i) Observations from (a)(i) to (a)(iii) can be used to identify one of the functional groups present in **FA 8**.

Identify the functional group and explain your answer, showing clearly your reasoning.

Functional group: ketone

Explanation: Since FA 8 undergoes condensation with 2,4-DNPH in (a)(iii), FA 8 contains either a ketone or aldehyde. However, since FA 8 does not undergo oxidation with Tollens' reagent in (a)(ii), it does not contain an aldehyde. [1]

M44	
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- (ii) Using observations from (a)(iv) to (a)(v), identify the other functional group present in **FA 8**.

Quote evidence from the relevant test to support your conclusion.

Functional group: alcohol

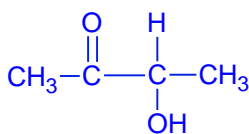
Evidence: Since FA 8 undergoes acid-metal displacement reaction with Na in (a)(v) to give H₂, it contains an alcohol functional group.

[1]

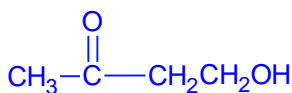
M45	
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- (iii) Suggest a possible structure of **FA 8** that are consistent with all the observations in **Table 4.1**.



OR



[1]

M46

(c) Inorganic analysis

FA 9 is an aqueous solution that contains a mixture of salts with two cations and one anion listed in the Qualitative Analysis Notes. One of the cations is M^{3+} in **FA 5**.

You should indicate clearly at what stage in a test a change occurs. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

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

No additional tests for ions present should be attempted.

Table 4.2

	tests	observations
(i)	Test the FA 9 solution using Universal Indicator paper.	<ul style="list-style-type: none"> • Universal indicator paper turns red OR orange pH 1 – 3
(ii)	<p>To 2 cm depth of FA 9, add aqueous sodium hydroxide dropwise with shaking till the test-tube is half-filled.</p> <p>Swirl and filter the mixture, collecting the filtrate in a test-tube. The filtrate is FA 10 which should be put to one side for use in (iii) to (v).</p>	<ul style="list-style-type: none"> • Red-brown ppt formed, insoluble in excess NaOH. • Red-brown residue & Colourless filtrate
(iii)	To 1 cm depth of FA 10 , carefully add nitric acid dropwise until no further change is seen.	<ul style="list-style-type: none"> • White ppt formed, • Ppt soluble in excess HNO_3 to give colourless solution • No gas/effervescence evolved

	tests	observations
(iv)	To 1 cm depth of FA 10 , add 1 cm depth of nitric acid, followed by silver nitrate. Then add aqueous ammonia slowly, with shaking, until no further change is seen.	<ul style="list-style-type: none"> • White ppt observed • Ppt soluble in excess NH_3 to give a colourless solution
(v)	To 1 cm depth of FA 10 , add 1 cm depth of nitric acid, followed by barium nitrate.	<ul style="list-style-type: none"> • No white ppt observed / No observable change

[3]

M47	M48	M49

- (d) (i) Explain your observations in (c)(i), given that one of the cations present in **FA 9** is M^{3+} .

M^{3+} has high charge density and is able to distort the electron cloud of the surrounding H_2O molecules, weakening and breaking the O-H bond, thus releasing H^+ . The hydrolysis of M^{3+} in water accounts for the acidic nature of the solution which turns the Universal Indicator paper red / orange.

[1]

M50	
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- (ii) From your observations in (c), suggest with evidence the identity of the anion present in **FA 10**.

Anion: Cl^-

Evidence: In test (c)(v), FA10 forms a white ppt of AgCl with AgNO_3 , white ppt soluble in excess NH_3 (aq) to give colourless solution.

[1]

M51	
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- (iii) Explain the observations in (c)(iii) when nitric acid was added to **FA 10**. Hence, suggest the identity of two possible cations that could be present in **FA 10**.

Addition of $\text{HNO}_3(\text{aq})$ neutralises the excess OH^- present in FA 10 causing the soluble complex $[\text{Al}(\text{OH})_4]^-$ / $[\text{Zn}(\text{OH})_4]^{2-}$ to form back the white ppt of $\text{Al}(\text{OH})_3$ / $\text{Zn}(\text{OH})_2$ respectively. In excess acid, the white ppt is neutralised and dissolve to form $\text{Al}^{3+}(\text{aq})$ / $\text{Zn}^{2+}(\text{aq})$.

Cations: Al^{3+} and Zn^{2+} [2]

M52	M53

- (iv) Based on the cations that you have identified in (d)(iii), devise a procedure to identify the cation in **FA 10**. Use a fresh sample of **FA 9** for this question and your tests should be based on the Qualitative Analysis Notes on pages 21–22 and should use only the bench reagents provided.

Record your tests and observations in the space below. Hence, state the identity of the cation in **FA 10**.

Any test requiring heating MUST be performed in a boiling tube.

Procedure	Observations
To 2 cm depth of FA 9 , add <u>aqueous ammonia dropwise</u> till <u>the test-tube is half-filled</u> .	<ul style="list-style-type: none"> Red-brown ppt formed, insoluble in excess NaOH.
<u>Swirl and filter the mixture</u> , collecting the <u>filtrate</u> in a test-tube.	<ul style="list-style-type: none"> Red-brown residue & colourless filtrate
To <u>1 cm depth of filtrate</u> , carefully add nitric acid dropwise until no further change is seen.	<ul style="list-style-type: none"> <u>White ppt formed, soluble in excess HNO_3 to give colourless solution</u> <u>Zn^{2+} is present</u>

[2]

M54	M55

[Total: 14]



Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	<i>reaction with</i>	
	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, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale 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 NO (pale) \rightarrow 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 acid)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acid)

(c) Test for gases

gas	tests 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

