TEMASEK JUNIOR COLLEGE

2022 JC2 PRELIMINARY EXAMINATIONS

HIGHER 2

CANDIDATE NAME	
CIVICS GROUP	/
CENTRE NO. / INDEX NO.	/

CHEMISTRY **9729/04**

Paper 4 Practical **29 August 2022**

2 hours 30 minutes

Candidates answer on the Question Paper.

DO NOT WRITE IN THIS MARGIN

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READ THESE INSTRUCTIONS FIRST

Write your Civics Group and name on all the work you hand in.
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 23 and 24.
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	/ 12
2	/ 19
3	/ 14
4	/ 10
Total	/ 55

This document consists of 22 printed pages and 2 blank pages.

[Turn over

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

1 Determination of the percentage by mass of water of crystallisation in $FeSO_4 \cdot nH_2O$

Read through the whole method before starting any practical work. Where appropriate, prepare a table for your results in the space provided.

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

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

$$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

In this experiment, you will determine the percentage by mass of water of crystallisation in $FeSO_4 \cdot nH_2O$. You will titrate a solution of **FA 1** against **FA 3**.

You are provided with

FA 1, contains 26.07 g dm^{-3} of hydrated iron(II) sulfate, $FeSO_4 \cdot nH_2O$, where n is an integer.

FA 2, 1.0 mol dm^{-3} dilute sulfuric acid, H_2SO_4.

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

You are to keep **FA 2** and **FA 3** for questions 2 and 3.

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(a) (i) Titration of FA 1 against FA 3

1. Fill a burette, labelled **FA 3**, with **FA 3**.
2. Use a pipette to transfer 10.0 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 2** to this flask.
4. Titrate **FA 1** with **FA 3** from the burette until the appearance of the first permanent pale-pink colour.
5. Record your titration results, to an appropriate level of precision, in the space on Page 3.
6. Repeat steps 1 to 4 until consistent results are obtained.

[Turn over

Titration Results

Final burette reading / cm^3	**9.20**	**9.30**
Initial burette reading / cm^3	**0.00**	**0.00**
Volume of FA 3 used / cm^3	**9.20**	**9.30**
	√	√

Tables have correct headers and units [1]
Note: Mark is lost if any final and initial burette readings are inverted or 50 is used as the initial burette reading.

All burette readings & volume used for all accurate titres in the titration table are recorded to 2dp and at least two uncorrected titres for end-point within ±0.10 cm^3. [1]

[2]

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

Average titre = $\frac{9.20 + 9.30}{2}$

= 9.25 cm^3 [1]

Answer should be recorded to 2.d.p

Mark is lost if there are arithmetic errors in the table.

Mark is lost if the titres used are not identified either in the table (by, for example, a tick) or in a calculation.

Accuracy
2 marks: If difference is ≤ 0.20cm^3

1 mark: If difference is ≤ 0.40 cm^3

0 mark: For a difference > 0.40 cm^3

*V*olume of **FA 3** = ……[3]

(b) **(i)** Calculate the amount of Fe^{2+} in 10.0 cm^3 of **FA 1**.

$5Fe^{2+} \equiv MnO_4^-$

No. of moles of Fe^{2+} = 9.25/1000 × 0.020 × 5

= • 9.25 x 10^{-4} mol [1]

amount of Fe^{2+} in 10.0 cm^3 of **FA 1** = …… [1]

(ii) Calculate the amount of Fe^{2+} in 1 dm^3 of **FA 1**.

No. of moles of Fe^{2+} = 9.25 x 10^{-4} x $\frac{1000}{10}$

= • 0.0925 mol [1]

amount of Fe^{2+} in 1 dm^3 of **FA 1** = ... [1]

(iii) Calculate the M_r of the hydrated iron(II) sulfate, $FeSO_4.nH_2O$, in **FA 1**.

M_r of $Fe(SO_4).nH_2O$ = 26.07 / 0.0925

= 281.8 [1]

M_r of the hydrated iron(II) sulfate = ...

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

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

n = {281.8 – [(55.8) + (32.1) + 4(16.0)]} ÷ 18.0

= 7.22

= 7 (nearest whole number) [1]

Note: This answer must be a positive integer.

n = ...

[2]

(iv) Hence, determine the percentage by mass of water of crystallisation in $FeSO_4 \cdot nH_2O$.

Show your working.

percentage by mass of water of crystallisation in $FeSO_4 \cdot nH_2O$.

= $\frac{7.22 \times 18}{281.8}$ x100%

= 46.1% [1]

Note for marker: If student uses the rounded off integer, need to re-calculate the M_r. otherwise, penalise.

Percentage by mass of water of crystallisation = ...

[1]

(v) Iron(II) sulfate in solution is readily oxidised by air to form iron(III) sulfate.

State the effect, on the value of *n* calculated in **(b)(iii)**, if some of your sample of **FA 1** had oxidised before you carried out the titration. Explain your answer.

Amount /Volume of MnO_4^- used is smaller OR mass/amount of Fe^{2+} used is smaller [1]

mass/moles/amount water larger so (mole) ratio larger so n increases [1]

Allow difference in titre/moles of MnO_4^- / Fe^{2+} will be too small to change the (integer) value of n for 1 mark.
Allow for 1 mark: less MnO_4^- / Fe^{2+} and n increases

[2]

[Total: 12]

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2 To investigate the reaction between manganate(VII) ions and $C_2O_4^{2-}$ ions.

In this experiment, you will investigate the kinetics of the reaction between MnO_4^- and $C_2O_4^{2-}$.

FA 4 is 0.0100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$

FA 5 is 0.200 mol dm^{-3} sodium ethanedioate, $Na_2C_2O_4$

FA 6 is 0.100 mol dm^{-3} potassium iodide, KI

FA 2 and **FA 3** are solutions from question 1.

You are to keep **FA 5** for question 3.

You are also provided with a starch indicator.

Acidified potassium manganate(VII) ions oxidises $C_2O_4^{2-}$ ions to produce Mn^{2+} ions which act as an autocatalyst for the reaction.

$$2\ MnO_4^-(aq) + 5\ C_2O_4^{2-}(aq) + 16\ H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

Acidified potassium manganate(VII) ions also oxidises I^- ions according to the following reaction:

$$2MnO_4^-(aq) + 10I^-(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$

You will add a solution of acidified potassium manganate(VII) to a mixture containing **FA 2** and **FA 5**. At timed intervals, you will draw 10 cm^3 aliquots of the reaction mixture. The

concentration of MnO_4^- ions in each aliquot will be determined after adding the aliquot to an excess of I^- solution and titrating the iodine produced against $S_2O_3^{2-}$ solution.

The reaction between iodine from the reaction mixture and $S_2O_3^{2-}$ solution is given below.

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

Each titration can only be performed once, so you must work very carefully.

Note: $C_2O_4^{2-}$ does not react with I^- in the reaction mixture.

In an appropriate format in the space provided on page 8, draw a table to record your

- transfer time (in minutes and seconds, to the nearest second) when half of the reaction mixture has emptied from the pipette,
- the decimal time, t_d, in minutes, to 0.1 min, for example, if t = 3 min 20 s then t_d = 3 min + 20/60 = 3.3 min,
- titration results for each of your aliquots. Make certain that your recorded results show the precision of your working.

Procedure

Note: Read all instructions in the procedure before you start performing the experiment.

1. Label each of the large test-tubes **1** to **6**. Using a measuring cylinder, add approximately 10 cm^3 of **FA 6** to each of these test-tubes.
2. Using appropriate measuring cylinders, transfer 50.0 cm^3 of **FA 5**, 5.0 cm^3 of **FA 2** and 45.0 cm^3 of deionised water into a conical flask labelled **reaction mixture**.
3. Using an appropriate measuring cylinder, transfer 25.0 cm^3 of **FA 3** to the conical flask labelled **reaction mixture**. Start the stopwatch and swirl the mixture thoroughly to mix its contents.
4. At approximately 1 minute, use a 10 cm^3 pipette to remove a 10.0 cm^3 aliquot of the reaction mixture. **Immediately** transfer this aliquot into the test-tube labelled **1** and swirl the mixture. Note and record the transfer time (in minutes and seconds, to the nearest second) when half of the reaction mixture has emptied from the pipette.
5. At approximately 2 minutes, repeat step 4. Transfer this aliquot into the large test-tube labelled **2**.
6. Repeat step 4 four more times at about three minute intervals, transferring the aliquots into test-tubes labelled **3** to **6**.

Note: Performing all of the titrations only after all the aliquots have been collected may affect time management.

[Turn over

Titrations

7. Pour the contents of test-tube labelled **1** into a clean 250 cm^3 conical flask. Rinse the test-tube and add the washings to the conical flask.

8. Titrate the iodine in this solution with **FA 4**. When the colour of the solution turns pale yellow, add about 1 cm^3 of starch indicator. The solution will turn dark blue/black. The end-point is reached when the dark blue/black colour just disappears. Record your results.

9. Repeat steps **8** to **9** for each of the remaining test-tubes labelled **2** to **6**.

(a) Experimental results

Specified Time	1 min	2 min	5 min	8 min	11 min	14 min
Transfer Time (in minutes and seconds)	**1 min 4 s**	**2 min 2 s**	**5 min 2 s**	**8 min 8 s**	**11 min 2 s**	**14 min 8 s**
Decimal Time, t_d /min	**1.1**	**2.0**	**5.0**	**8.1**	**11.0**	**14.1**
Final Burette Reading /cm^3	**19.70**	**19.70**	**17.35**	**27.30**	**30.80**	**32.60**
Initial Burette Reading /cm^3	**0.00**	**0.00**	**0.00**	**17.50**	**27.50**	**31.00**
Volume of **FA 4** added /cm^3	**19.70**	**19.70**	**17.35**	**9.80**	**3.30**	**1.60**

[3]

Table with correct headers and units **[1]**

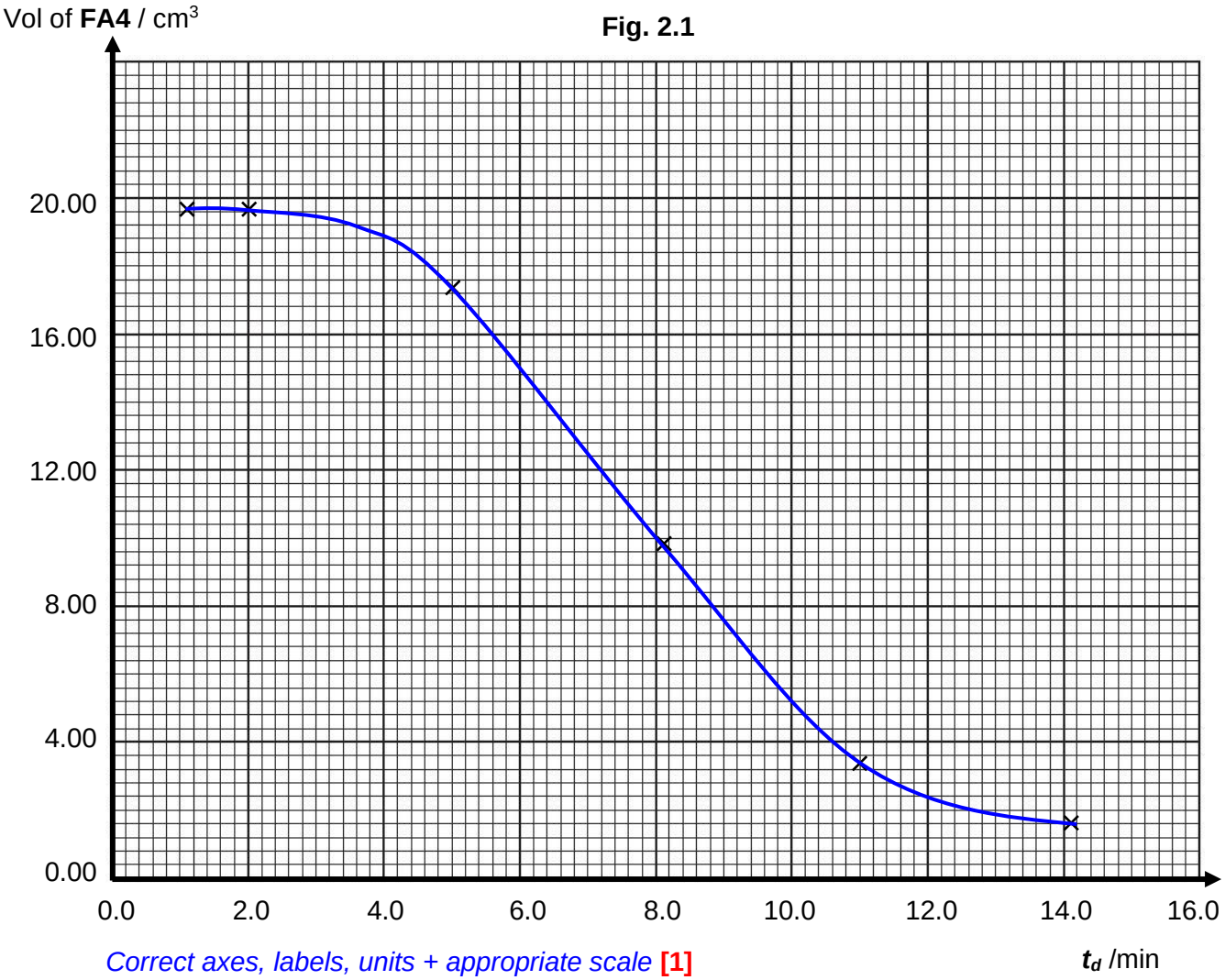
6 sets of results of correctly calculated values and interval for transfer time **[1]**

Note: results should be within 30 s of specified time

1 dp for decimal time **[1]**

(b) **(i)** On the grid in **Fig. 2.1**, plot a graph of **volume of sodium thiosulfate**, **FA 4**, on the *y*-axis, against time, t_d, on the *x*-axis. You should draw the most suitable line that takes into account all of your plotted points.

Fig. 2.1



Correct axes, labels, units + appropriate scale **[1]**

All points correctly plotted **[1]**

Smooth curve and correct shape showing a gentler gradient at the start, gradient becomes steeper with time **[1]**

(ii) Consider the **shape** of the graph in **Fig.2.1.**

Describe the shape and explain how it relates to the rate of reaction between MnO_4^- and $C_2O_4^{2-}$.

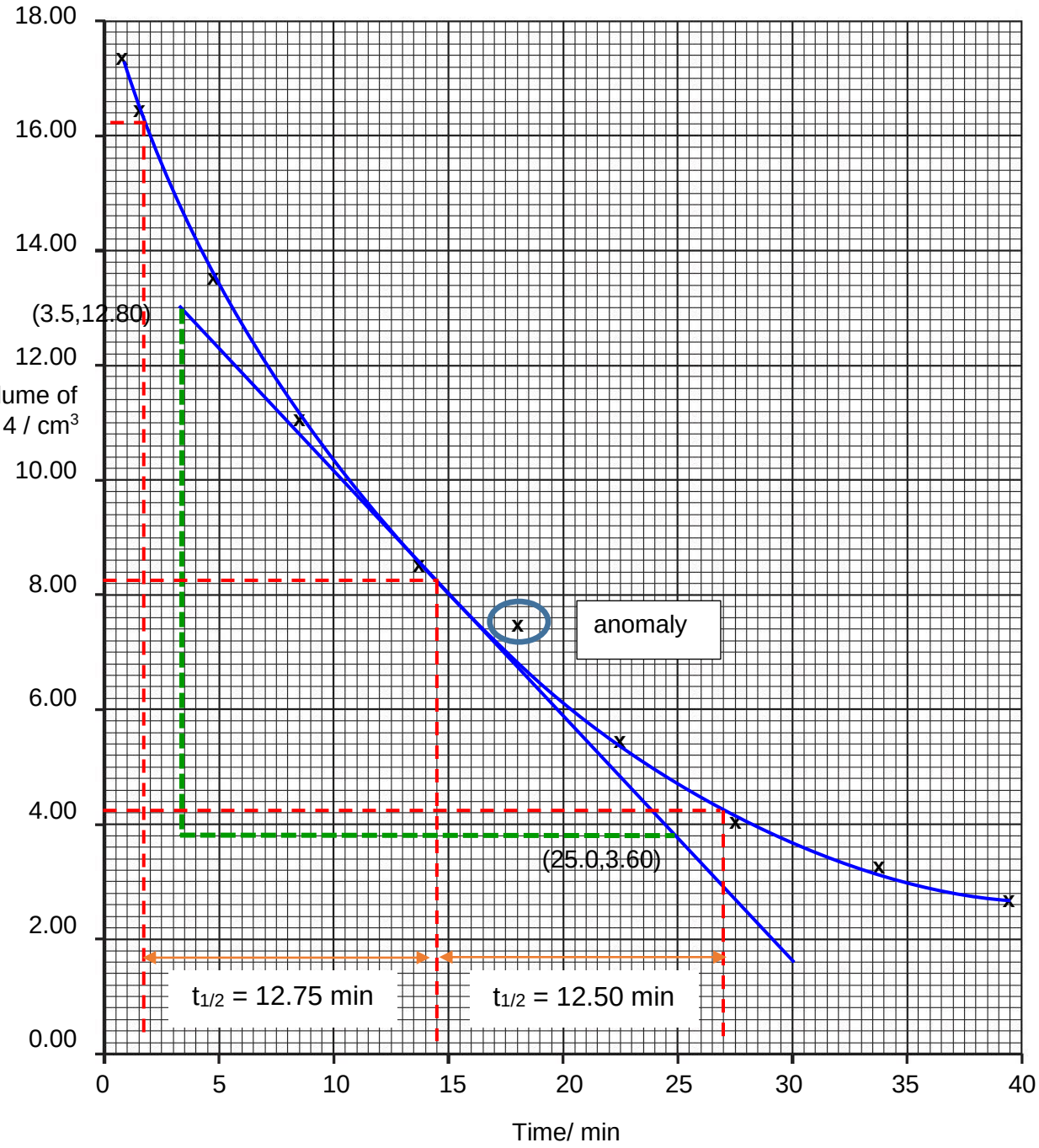
Initially, the almost constant gradient shows that the reaction starts slowly as very little Mn^{2+} is formed to catalyse reaction. Rate of reaction then increases, as seen from the steeper gradient, since more Mn^{2+} are produced to catalyse the reaction. Finally, rate of reaction decreases as seen from the less steep gradient since the concentration of reactants decrease as reaction proceeds. **[1]**

[1]

2 **(c)** A student performed a similar experiment under a different temperature. In step **2** of the procedure on page 8, she used the same volumes of **FA 2** and **FA 5** that you used but she also added 5.0 cm^3 of 1.00 mol dm^{-3} manganese(II) sulfate. To keep the total volume of the same as your experiment, she added 40.0 cm^3 of deionised water.

The data from the student's experiment has been plotted in **Fig. 2.2**.

(i) Draw the most appropriate line taking into account all of the plotted points. [1]



Comment: draw a smooth best fit curve, ignoring the anomalous point at 18 min.

(ii) Using the graph in **(c)(i)**, calculate the [MnO_4^-] in the reaction mixture at ***t*** = 15 min. [2]

Volume of $S_2O_3^{2-}$ = 7.90 cm^3 [1]

Reminder of page 6 procedure:

*At timed intervals, you will draw **10 cm^3 aliquots** of the reaction mixture. The concentration of MnO_4^- ions in **each (10 cm^3) aliquot** will be determined after adding the aliquot to an excess of I^- solution and titrating the iodine produced against $S_2O_3^{2-}$ solution.*

No. of moles of $S_2O_3^{2-}$ = 7.90 x 10^{-3} x 0.0100 = 7.90 x 10^{-5} mol

MnO_4^- ≡ 5 $S_2O_3^{2-}$

No. of moles of MnO_4^- = 7.90 x 10^{-5} ÷ 5 = 1.58 x 10^{-5} mol

[MnO_4^-] = 1.58 x 10^{-5} ÷ (10 x 10^{-3}) = 1.58 x 10^{-3} mol dm^{-3} [1]

Note: each volume of FA4 sampled is based on only 10 cm^3 solution pipetted out, NOT the whole solution of 100 cm^3.

(iii) Draw a tangent to your graph line in **(c)(i)** at ***t*** = 15 min. Calculate the gradient of this line, and hence determine the rate of change of the **amount** of $S_2O_3^{2-}$ ions required in mol min^{-1}. [3]

Gradient = – (12.80-3.60)/(3.5-25.0) = –0.4278 cm^3 min^{-1} [1]

Drawing of tangent on graph and calculation of gradient using co-ordinates that are 3 big squares in both x and y direction [1]

Note: read off coordinates from the graph carefully. Gradient coordinates should be as wide as possible (at least 3 big sq in both x and y direction).

Note: gradient represents rate of change of volume of $S_2O_3^{2-}$ ions

Rate of change of amount of thiosulfate ions = 0.428 x 10^{-3} x 0.0100

= 4.28 x 10^{-6} mol min^{-1} [1]

(iv) Using your answer in **(c)(iii)**, calculate the rate of change of MnO_4^- in mol min^{-1}. [1]

2 MnO_4^- ≡ 5 I_2 ≡ 10 $S_2O_3^{2-}$

MnO_4^- ≡ 5 $S_2O_3^{2-}$

rate of change of MnO_4^- = 4.28 x 10^{-6} ÷ 5 = 8.56 x 10^{-7} mol min^{-1} [1]

(v) Hence, calculate the rate of disappearance of [MnO_4^-] in the reaction mixture, at time ***t*** = 15 min. [3]

rate of disappearance of [MnO_4^-] = 8.56 x 10^{-7} ÷ (10 x 10^{-3})

= 8.56 x 10^{-5} mol dm^{-3} min^{-1} [1]

Note: only 10 cm^3 of solution containing $KMnO_4$ is extracted for titration each turn, NOT the whole solution of 100 cm^3.

Shows working in all calculations. All calculations must be relevant although they may not be complete or correct. Any calculation not attempted loses this mark. [1]

Shows appropriate significant figures (3 or 4 sf) and units in all final answers. Any calculation not attempted loses this mark. [1]

(d) Using the graph in **(c)(i)**, show that the reaction between MnO_4^- and $C_2O_4^{2-}$ is first order with respect to [MnO_4^-]. [2]

Showing the construction lines for half-life [1] for at least 2 similar half-lives. Do NOT find half-life from t = 0 min as initial volume of FA4 used is not shown.

The volume of FA 4 used is directly proportional to the no. of moles of I_2 liberated by the unreacted MnO_4^- from the reaction. Hence, the volume of FA 1 α [MnO_4^-]. Since a constant half-life = 12.50 min, is obtained from the graph, the order of reaction with respect to [MnO_4^-] is 1.

Note: relationship between FA4 and [$KMnO_4$] must be clearly shown

→ [$KMnO_4$] ↓ as rxn progresses → [I_2] ↓ → [$S_2O_3^{2-}$] ↓

explanation [1]

[Total: 19]

3 To explore the chemistry of some compounds of unknown transition elements X and a hydrocarbon.

FA 7 (MnO_2) is a solid sample of a common dioxide of the unknown transition element **X**

FA 8 (Mn^{2+}, SO_4^{2-}) is a pure solution of compound **Y,** which is the product formed in **3(a)**

FA 9 (cyclohexene) is a hydrocarbon

You will need access to the **FA 2**, **FA 3** and **FA 5** from question 1 and 2.

Perform the tests described in Table 3.1 and record your observations in the table. Test and identify any gases evolved. The observation in **(c)(iii)** has been completed for you. There is no need to carry out this test.

Table 3.1

tests		observations
(a)	Add 2 cm depth of **FA 2** (H_2SO_4) and 2 cm depth of **FA 5** ($Na_2C_2O_4$) to a test-tube.	
	Using a spatula, add **FA 7** (MnO_2) to the mixture in small portion with swirling until no further change.	√ **Effervescence** √ **Colourless, odourless, acidic gas gave white ppt with limewater**
	Filter the mixture into a test-tube. The filtrate contains compound **Y**.	√ **Pale pink/colourless/pale yellow/ yellow-green filtrate and black residue (remaining unreacted FA 7)**
	Divide the filtrate into two portions. To one portion, add aqueous sodium hydroxide until no further change.	√ **Light brown ppt, insoluble in excess NaOH(aq). Ppt darkens on standing**
	To the other portion, add aqueous ammonia until no further change.	√ **Light brown ppt, insoluble in excess NH_3(aq). Ppt darkens on standing**
(b)(i)	Place 1 cm depth of **FA 8** in a test-tube, add aqueous sodium hydroxide.	√ **Off-White ppt/very light brown ppt, ppt darken on standing / rapidly turning brown on contact with air, insoluble in excess**
(b)(ii)	Place 1 cm depth of **FA 8** in a test-tube, add aqueous ammonia solution.	√ **Off-White ppt/very light brown ppt, ppt darken on standing / rapidly turning brown on contact with air, insoluble in excess** Note: the colour of ppt in (b) is lighter than ppt observed in (a)

(c)(i)	Place 5 cm depth of aqueous sodium hydroxide in a test-tube. Add **5 drops** of **FA 9** to this test-tube. Add **FA 3,** dropwise with shaking, until no further change is seen. Allow to stand for 5 minutes, with occasional shaking. Continue with the remaining parts of Question 3.	√ **Solution turns green/blue green** √ **Colour deepens as more FA 3 added** √ **Brown ppt formed**
(c)(ii)	Place 5 cm depth of **FA 2** in a test-tube. Add **5 drops** of **FA 9** to this test-tube. Add **FA 3,** dropwise with shaking, until no further change is seen. Allow to stand for 5 minutes, with occasional shaking. Continue with the remaining parts of Question 3.	√ **Purple $KMnO_4$ decolourises.** √ **Solution turns pink,** √ **then turns brown/ orange/ decolourises.**
(c)(iii)	Place 5 cm depth of water in a test-tube. Add **1 drop** of **FA 9** to this test-tube. Add bromine water, dropwise with shaking.	**Bromine water decolourised**

[6]

[The points are scaled to 6 marks as follows:

12-13 √ = 6; 10 – 11 √ = 5; 8-9 √ = 4; 7-6 √ = 3 ; 5-4 √ = 2; 2-3= 1]

(d) Consider your observations in **(a)** and **(b)**, identify the transition ion formed in **(a)**. Justify your choice by referring to the observations in **(b)**.

Ion present is **Mn^{2+}**

Justification: **off-white ppt formed with both NaOH and NH_3, ppt insoluble in excess and darken on standing**

[1]

(e) The anion in **FA 8** is not a nitrate, nitrite or carbonate ion.

You will devise and perform a series of simple tests to identify the anion present in **FA 8**.

Use the Qualitative Analysis Notes on pages 23-24 to deduce the possible anions in **FA 8** and describe **two different** tests, that will allow you to distinguish between the possible anions. You can only use the bench reagents provided.

Perform the tests described and record your observations in the space below. Hence, deduce the identity of the anion in **FA 8.**

Possible anions: **[√] SO_3^{2-}, SO_4^{2-}, I^-, Cl^-, Br^-**

Tests	Observation
To 1 cm depth of FA 8 in a test tube, add $AgNO_3$(aq) followed by dilute HNO_3(aq). [√]	**No ppt formed. [√]**
To 1 cm depth of FA 8 in a test tube, add $Ba(NO_3)_2$(aq) followed by dilute HNO_3(aq). [√]	**White ppt formed** **Insoluble in nitric acid [√]**

Identity of anion: **[√] SO_4^{2-}**

2 √: 1 mark

[3]

(f) Compound **Z** is the main organic product in **(c)(i)** when **FA 9** reacts with **FA 3** under alkaline conditions. The molecular formula of **Z** is $C_6H_{12}O_2$.

(i) Deduce the molecular formula of **FA 9**.

Explain your deduction. Your explanation should be supported by evidence from your observations in **(c)**.

Molecular formula of **FA 9** is **C_6H_{10} [1]**

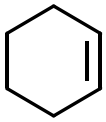
Explanation: **[1]**

- **Br_2(aq) decolourised, C=C present**
- **Cold alkaline MnO_4^- oxidises C=C to 1,2-diol C(OH)-C(OH)**
- **Only two oxygen atoms in molecular formula of Z, so only one C=C bond**
- **FA 9 is $C_6H_{12}O_2$ – 2OH = C_6H_{10}**

[2]

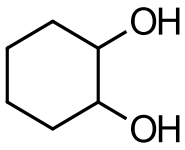
(ii) Draw the structural formulae for **FA 9** and compound **Z**.

structure of **FA 9**



[1]

structure of **Z**



[1]

[2]

[Total:14]

4 Planning

You are to plan an experiment to determine the concentration of a solution of $H_2C_2O_4$ and the ΔH_{neut} between ethanedioic acid and sodium hydroxide.

$$\tfrac{1}{2}\ H_2C_2O_4(aq) + NaOH(aq) \rightarrow \tfrac{1}{2}\ Na_2C_2O_4(aq) + H_2O(l) \qquad \Delta H_{neut}$$

You are provided with 2.00 mol dm^{-3} NaOH and a solution of $H_2C_2O_4$ of approximately the same concentration. A series of experiments can be carried out using different volumes of the two solutions. In each experiment, the total volume of the two solutions is to be kept at 50 cm^3 and the increase in temperature, ΔT, is determined.

A graph of ΔT against *volume of* $H_2C_2O_4$ can then be used to determine the exact concentration of the $H_2C_2O_4$ solution and ΔH_{neut}.

(a) Determine the volume of $H_2C_2O_4$ and of NaOH to be used such that both solutions will react completely. Show your working clearly and give your answers to the **nearest whole number**.

$$\frac{[H_2C_2O_4] \times \text{Volume } H_2C_2O_4}{[NaOH] \times \text{Volume NaOH}} = \frac{1}{2}$$

Since $[H_2C_2O_4] \approx [NaOH]$,

$\Rightarrow$ Volume $H_2C_2O_4$: Volume NaOH = 1:2

Volume of $H_2C_2O_4$ = 50 × 1/3 = 17 cm^3

Volume of NaOH = 50 × 2/3 = 33 cm^3

OR

No. of moles of $H_2C_2O_4$ = V x $[H_2C_2O_4]$

Vol. of NaOH = (2V x [$H_2C_2O_4$]) / [NaOH] = 2V (since [$H_2C_2O_4$] ≈ [NaOH])

2V + V = 50 (since total vol of mixture = 50 cm^3)

V = 16.7 cm^3 (~ 17 cm^3) = vol of $H_2C_2O_4$

Volume of NaOH = 2 x 16.7 = 33.3 cm^3 (~ 33 cm^3)

for both volumes, including working [1]

[1]

(b) Plan a procedure to collect sufficient data to allow a graph of ΔT against *volume of* $H_2C_2O_4$ to be drawn.

You are provided with:

- 2.00 mol dm^{-3} NaOH,
- $H_2C_2O_4$ solution of approximately the same concentration,
- the equipment normally found in a school laboratory.

In your plan, you should include the following:

- the procedure, including the apparatus you would use
- a tabulation of the experimental data to be collected, including the volumes of $H_2C_2O_4$ and NaOH

You may assume that the $H_2C_2O_4$ and NaOH solutions have the same initial temperature.

1. **Use a 10 cm^3 measuring cylinder to measure 5 cm^3 of $H_2C_2O_4$ (or use a 50 cm^3 measuring cylinder to measure 45 cm^3 NaOH) and add it into a styrofoam cup. Place this styrofoam cup into a second styrofoam cup, which is then placed in a beaker for support.**
2. **Use a thermometer to measure and record the initial temperature of the solution in the cup.**
3. **Use a second 50 cm^3 measuring cylinder to measure 45 cm^3 of NaOH (or Use a 10 cm^3 measuring cylinder to measure 5 cm^3 $H_2C_2O_4$) and add this second solution to the first solution in the Styrofoam cup.**
4. **Use the thermometer to stir the mixture and measure the highest temperature reached.**
5. **Wash and dry the styrofoam cup and the thermometer.**
6. **Repeat the above steps using the volumes as shown in the table.**

Volume of $H_2C_2O_4$ /cm^3	Volume of NaOH /cm^3	Initial temperature of $H_2C_2O_4$ /°C	Highest temperature after mixing /°C	ΔT /°C
5 10 15	45 40 35			
20 30 40	30 20 10			

- **overall method [1]**

  Do not award this mark if student added water / uses the same volume of $H_2C_2O_4$ or NaOH / uses a burette to add the second solution to the first solution / planned an experiment to take a temperature readings at 1 min, 2 min, 3 min, 4 min, 5 min etc.

- **suitable choice of apparatus (thermometer, measuring cylinders, Styrofoam cup) [1]**

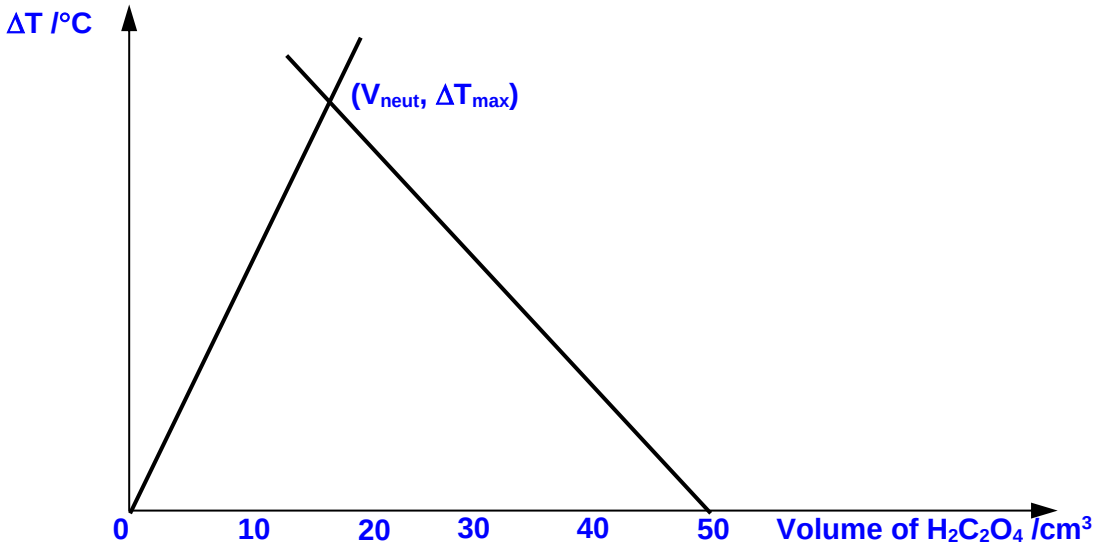
  Note: '10 cm^3 measuring cylinder' is not suitable for larger volumes. 50 cm^3 measuring cylinders would be more appropriate.

- **suitable volumes of $H_2C_2O_4$ and NaOH (Total volume must be 50 cm^3 with at least three $H_2C_2O_4$ volumes < 17 cm^3 and at least three > 17 cm^3 so that there are at least three plotted points on each line) [1]**

- **table that recorded the initial temperature of $H_2C_2O_4$ (or NaOH) and the highest temperature reached after mixing. [1]**

[4]

(c) Using your answer in **(a)**, sketch, on **Fig. 4.1**, the graph you would expect to obtain from the results.



[Turn over

• two straight lines that intersect, inverted V-shape,

• must start from 0 cm^3, intersection point shows volume $H_2C_2O_4$ more to the left because it is around 17 cm^3 or around 1/3), second line should intersect x-axis if V_{acid} = 50cm^3 / should not intersect if V_{acid} < 50cm^3

(d) Suggest a method to minimise heat lost to the surroundings to ensure the results obtained are accurate.

Use a lid for the styrofoam cup / switch off the fans to ensure draft-free environment / using *two* styrofoam cups stacking one into the other [1]

[1]

(e) Another student conducted a similar experiment but used 1.00 mol dm^{-3} of NaOH instead. He obtained the following results:

- Volume of ethanedioic acid needed for complete neutralisation = 15 cm^3
- Maximum temperature change = 9.0°C

Calculate the concentration of the $H_2C_2O_4$ solution, in mol dm^{-3} and the ΔH_{neut} in kJ mol^{-1}.

You may assume the following:

- specific heat capacity of all solutions is 4.18 J g^{-1} $°C^{-1}$,
- density of all solutions is 1.00 g cm^{-3}.

No. of moles of NaOH = $\frac{50-15}{1000}$ x 1

= 0.0350 mol

$H_2C_2O_4$ Ξ 2NaOH,

$[H_2C_2O_4]$ = (½ x 0.035)/(15/1000)

= 1.17 mol dm^{-3} [1]

No. of moles of H_2O produced = no. of moles of NaOH reacted

= 0.0350 mol

$\Delta H_{neut} = -\frac{(50 \times 4.18 \times 9.0)/1000}{0.0350}$

= - 53.7 kJ mol^{-1} [1]

[2]

[Total: 10]

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 → (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) Colours 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