



TAMPINES MERIDIAN JUNIOR COLLEGE

JC2 PRELIMINARY EXAMINATION

CANDIDATE NAME

Answers

CIVICS GROUP

H2 CHEMISTRY

Paper 4 Practical

9729/04

3 September 2025

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 21 and 22.

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

Shift

Laboratory

For Examiner's Use

1 / 10

2 / 19

3 / 17

4 / 9

Total / **55**

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

1 Qualitative analysis tests involving solid iron(III) chloride

FA 1 is a solution containing iron(III) chloride, $\text{FeCl}_3 \cdot n\text{H}_2\text{O}$

FA 2 is a solution containing an unknown L^- ligand

Carry out the following tests. Record your observations in Table 1.1.

Unless otherwise stated, the volumes given in Table 1.1 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**.

Table 1.1

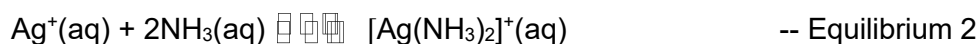
| | | Test | Observations | Explanation (teacher's notes) |
|-----|-------|---|--|---|
| (a) | (i) | Add about 6 cm depth of FA1 into a test tube. Add aqueous silver nitrate in excess. Filter the mixture. Separate the filtrate into four test tubes for (iii), (iv), (v) and (vi). | <u>white ppt formed in yellow/orange solution</u> <u>White residue/ppt obtained</u> <u>Yellow/orange filtrate/solution obtained</u> | Cl^- is present to form AgCl ppt $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ complex is present |
| | (ii) | Place the filter funnel containing the residue on a new test tube, add aqueous ammonia over the residue and collect the filtrate. To this filtrate, add nitric acid dropwise until no further changes. | The white ppt was <u>soluble/dissolves in aqueous ammonia to form a colourless/pale yellow/orange solution.</u> With nitric acid, <u>white ppt formed. White ppt remains insoluble in excess nitric acid.</u> | Cl^- is present. $[\text{Ag}(\text{NH}_3)_2]^+$ is formed. It should be a colourless solution but the ppt is covered with traces of Fe^{3+} , hence the solution appears pale yellow. Reversal of equilibrium. HNO_3 neutralise the NH_3 and hence AgCl is formed again. |
| | (iii) | To the first test tube containing the filtrate from (i), add 2 cm^3 of hydrogen peroxide. Observe the mixture until no further changes are seen. | <u>Brown/orange solution formed.</u> <u>Effervescence observed. Gas evolved relights a glowing splint. O_2 (g) is evolved.</u> <u>Brown solution turns back to yellow/orange on standing</u> | O_2 evolved. FA 2 acts as a reducing agent; Fe^{3+} acts as an oxidising agent. |



| | | | | |
|--|------|--|---|--|
| | (iv) | To the second test tube containing the filtrate from (i), add an equal volume of aqueous sodium hydroxide. Then add 2 cm ³ of hydrogen peroxide. Observe the mixture until no further changes are seen. | <u>Red-brown ppt formed</u> <u>(More) effervescence observed. Gas evolved relights a glowing splint. O₂(g) is evolved.</u> | Fe(OH) ₃ is produced. More O ₂ released as H ₂ O ₂ exists as HO ₂ ⁻ and have a greater reducing ability (gets oxidised more easily by remaining Fe ³⁺). Solid Fe(OH) ₃ does not undergo reduction |
| | (v) | To the third test tube containing the filtrate from (i), add 1 cm ³ of FA 2. | <u>(Blood) red/black solution formed.</u> | [Fe(H ₂ O) ₆] ³⁺ undergoes a ligand exchange reaction to form [Fe(SCN)] ²⁺ . |
| | (vi) | To the last test tube containing the filtrate from (i), add half a spatula of iron filings. Then add an equal volume of hydrochloric acid. Dispose the reaction mixture once observations are completed to prevent gas build up. | <u>No observable change (when Fe is added)</u> <u>Effervescence observed</u> <u>Gas evolved "pops" with a lighted splint. H₂(g) is evolved</u> <u>Yellow/orange solution turns light green/colourless</u> | Comproportionation reaction occurred. Fe is oxidised to Fe ²⁺ , while Fe ³⁺ is reduced to Fe ²⁺ , as shown by the colour change (Fe ²⁺ (aq) is green) |

[5]

- (b) (i) With the aid of the following equilibrium equations, explain the observations obtained in Test (ii) when nitric acid was added to the filtrate.



The white solid residue is AgCl. When NH₃(aq) is added, the colourless and soluble silver complex [Ag(NH₃)₂]⁺ is formed in the filtrate.

When HNO₃ is added, the H⁺ from the acid neutralises NH₃(aq), resulting in equilibrium 2 to shift left, hence reproducing Ag⁺ and the white solid AgCl in equilibrium 1 again.

.. [1]



- (ii) In the presence of a strong base, hydrogen peroxide reacts with OH^- to form HO_2^- .

| electrode reaction | E^\ominus/V |
|--|---------------|
| $\text{Fe}^{3+} + \text{e}^- \rightleftharpoons \text{Fe}^{2+}$ | +0.77 |
| $\text{Fe}(\text{OH})_3 + \text{e}^- \rightleftharpoons \text{Fe}(\text{OH})_2 + \text{OH}^-$ | -0.56 |
| $\text{O}_2 + 2\text{H}^+ + 2\text{e}^- \rightleftharpoons \text{H}_2\text{O}_2$ | +0.68 |
| $\text{H}_2\text{O}_2 + 2\text{H}^+ + 2\text{e}^- \rightleftharpoons 2\text{H}_2\text{O}$ | +1.77 |
| $\text{O}_2 + \text{H}_2\text{O} + 2\text{e}^- \rightleftharpoons \text{HO}_2^- + \text{OH}^-$ | -0.08 |

With reference to the electrode potential given above, calculate the overall E^\ominus values for the reactions in Tests (iii) and (iv) respectively.

Hence, account for the difference in the rate of effervescence produced when hydrogen peroxide was added in Tests (iii) and (iv).

$$E^\ominus \text{ for (iii)} = 0.77 - 0.68 = +0.09 \text{ V}$$

$$E^\ominus \text{ for (iv)} = 0.77 - (-0.08) = +0.85 \text{ V}$$

($\text{Fe}(\text{OH})_3(\text{s})$ does not undergo reduction. If $\text{Fe}(\text{OH})_3$ values are used, the resulting E^\ominus values will be negative, which goes against any observable reaction.)

The effervescence is more vigorous for step (iv)

The overall E^\ominus value is more positive for step (iv) when hydrogen peroxide reacts with Fe^{3+} in alkaline medium, representing a more spontaneous reaction.

OR As the $E^\ominus (\text{O}_2/\text{HO}_2^-)$ is less positive than that of $E^\ominus (\text{H}_2\text{O}_2/\text{H}_2\text{O})$, HO_2^- is a stronger reducing agent than H_2O_2 [2]

- (iii) Based on your observation in Test (v), state the type of reaction that has occurred.

Ligand exchange reaction

[1]

- (iv) In Test (vi), a comproportionation reaction occurred, where two species of the same element undergoes a redox reaction to form the same product. Write an ionic equation to represent the reaction.



[1]

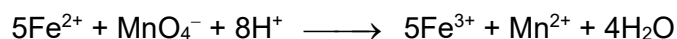
[Total: 10]



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

Iron(III) chloride solutions are often used as a coagulant to remove impurities in sewage and industrial waste.

FA 3 is hydrated iron(III) chloride with the formula $\text{FeCl}_3 \cdot n\text{H}_2\text{O}$. The addition of excess zinc to a solution of **FA 3** converts the Fe^{3+} ions to Fe^{2+} ions. The amount of Fe^{2+} ions can then be determined quantitatively by titration against a standard solution of potassium manganate(VII), KMnO_4 . The reaction between MnO_4^- and Fe^{2+} is as shown below.



In this experiment, you are to prepare a standard solution using **FA 3** and perform titrations to determine the value of n , the water of crystallisation in **FA 3**.

You are provided with the following:

FA 3 is solid hydrated iron(III) chloride, $\text{FeCl}_3 \cdot n\text{H}_2\text{O}$

FA 4 is $0.500 \text{ mol dm}^{-3}$ dilute sulfuric acid, H_2SO_4

FA 5 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4

Zinc powder

(a) Preparation of standard solution of hydrated iron(III) salt

1. Weigh the capped container with **FA 3**. Record the mass.
2. Transfer all the **FA 3** measured into a 250 cm^3 beaker. Reweigh the weighing bottle and record its mass. Determine and record the mass of **FA 3** transferred.
3. Use a 100 cm^3 measuring cylinder to add 100 cm^3 of **FA 4** to the beaker. Stir the mixture with a glass rod to dissolve all the solid.
4. Transfer the solution into a 250 cm^3 volumetric flask. Rinse the beaker with deionised water and pour the washings into the volumetric flask.
5. Make up to the 250 cm^3 mark with deionised water, stopper and mix thoroughly by inverting the flask several times.
6. Label the resultant solution **FA 6**.

(b) Preparation of Fe^{2+} solution from **FA 6**

7. Use a 100 cm^3 measuring cylinder to transfer 100 cm^3 of **FA 6** into a 250 cm^3 beaker.
8. Add all the zinc powder in the container into the beaker. Continuously stir the reaction mixture for 5 minutes.
9. Filter the mixture into a **dry** 250 cm^3 conical flask provided using **dry** filter paper and filter funnel. Ignore any reaction that may still be taking place.
10. Label the filtrate as **FA 7**. Proceed to (c) once you have collected sufficient filtrate.



(c) Titration of FA 7 against FA 5

11. Fill the burette with **FA 5** solution.
12. Use a 10 cm³ pipette to transfer 10.0 cm³ of **FA 7** into a 250 cm³ conical flask.
13. Use a 10 cm³ measuring cylinder to add 10.0 cm³ of **FA 4** to this flask.
14. Titrate the reaction mixture in the conical flask with **FA 5** from the burette until the appearance of the first permanent orange colour.
15. Record your titration results, to an appropriate level of precision, in the space below.
16. Repeat steps 12 to 15 until consistent results are obtained.

Results

| | |
|---|--------|
| Mass of weighing bottle and FA 3 / g | 11.280 |
| Mass of weighing bottle and residual FA 3 / g | 4.780 |
| Mass of FA 3 used / g | 6.500 |

| | | |
|---|------|------|
| Final burette reading / cm ³ | 9.60 | 9.60 |
| Initial burette reading / cm ³ | 0.00 | 0.00 |
| Volume of FA 5 used / cm ³ | 9.60 | 9.60 |

✓

✓

[4]



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

$$\begin{aligned}\text{Average volume of FA 5} &= \frac{9.60 + 9.60}{2} \\ &= 9.60 \text{ cm}^3\end{aligned}$$

volume of **FA 5** = [3]

- (ii) Calculate the amount of Fe^{2+} in 10.0 cm^3 of **FA 7**.



$$\begin{aligned}\text{No. of moles of Fe}^{2+} &= 9.60/1000 \times 0.020 \times 5 \\ &= \underline{9.60 \times 10^{-4} \text{ mol}}\end{aligned}$$

amount of Fe^{2+} in 10.0 cm^3 of **FA 7** = [1]

- (iii) In step 8, an excess of zinc was added to convert the Fe^{3+} to Fe^{2+} . Calculate the amount of Fe^{3+} in 250 cm^3 of **FA 6**.



$$\begin{aligned}\text{Amount of Fe}^{3+} &= 9.60 \times 10^{-4} \times \frac{250}{10} \\ &= \underline{0.0240 \text{ mol}}\end{aligned}$$

amount of Fe^{3+} in 250 cm^3 of **FA 6** = [1]



- (iv) Use your answer from (iii) to calculate the M_r of the hydrated iron(III) chloride, $\text{FeCl}_3 \cdot n\text{H}_2\text{O}$, in **FA 3**.



$$\text{Amount of } \text{FeCl}_3 \cdot n\text{H}_2\text{O} = 0.0240 \text{ mol}$$

$$M_r \text{ of } \text{FeCl}_3 \cdot n\text{H}_2\text{O} = 6.50 / 0.0240$$

$$= \underline{270.8}$$

$$M_r \text{ of the hydrated iron(III) chloride} = \dots\dots\dots$$

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

[A_r : Fe, 55.8; Cl, 35.5; O, 16.0; H, 1.0]

$$n = (270.8 - [55.8 + 3(35.5)]) \div 18.0$$

$$= \underline{6} \text{ (nearest whole number)}$$

$$n = \dots\dots\dots [5]$$

- (d) In step 10, excess zinc was filtered off before the titration of **FA 7** against **FA 5** to avoid any possible reaction between zinc and **FA 5**.

Suggest another reason why it was necessary to filter off the excess zinc metal, and what effect failing to do so would have on the titre values.

Zinc metal that is not removed will continue to reduce Fe^{3+} formed during the titration to Fe^{2+} , resulting in a higher than expected titre.

.....
.....

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



- (e) A student carried out the experiment and obtained a **FA 5** titre volume of 10.00 cm³ for the end-point. The absolute uncertainties associated with some of the apparatus used in the experiment are given below.

| | |
|-------------------------------|-----------------------|
| 10.00 cm ³ pipette | ±0.02 cm ³ |
| 50.00 cm ³ burette | ±0.05 cm ³ |

- (i) Use the data above to calculate the percentage uncertainty associated with each apparatus.

$$\% \text{ uncertainty in pipette} = \frac{0.02}{10.0} \times 100\% = \underline{\underline{0.200\%}}$$

$$\% \text{ uncertainty in burette} = \frac{0.05 + 0.05}{10.00} \times 100\% = \underline{\underline{1.00\%}}$$

[1]

- (ii) The percentage uncertainty associated with the use of the measuring cylinder is 2.00 %. Suggest why the percentage uncertainty associated with the use of the measuring cylinder is not considered when determining the overall percentage uncertainty of the experimental procedure.

Measuring cylinder is not considered as **FA 4 (sulfuric acid) is added in excess.**

[1]

- (f) (i) Suggest how the titration would be affected if 10.0 cm³ of water was added instead of **FA 4** in step 13.

If water is used, there will be **insufficient acid** which result in the reduction of MnO₄⁻ to form **brown ppt of MnO₂**. Hence, the **end point of the titration cannot be detected.**

[1]

- ((ii) Hydrogen peroxide is also able to oxidise the Fe²⁺ ions in an acidic medium. However, hydrogen peroxide is not a suitable replacement for potassium manganate in this experiment. Suggest a reason for this.

There is **no distinct/ visible/ significant colour change at the end-point.** Hence, it would be impossible to know when the reaction is completed.

OR

H₂O₂ undergoes **decomposition readily**, hence **titrant volume will be inconsistent.**

OR

Fe²⁺ or Fe³⁺ (small amount) present in the solution may **react with H₂O₂**

OR **Fe²⁺ or Fe³⁺ catalyses the decomposition of H₂O₂.**

OR **H₂O₂ can reduce Fe³⁺ formed in the titration.**

[Turn Over]

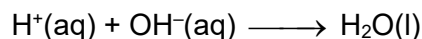


3 Determination of the enthalpy change of neutralisation, ΔH_{neut} between a strong acid, H_aX and NaOH(aq) .

FA 8 is 1.00 mol dm^{-3} of strong acid, H_aX .

FA 9 is 1.60 mol dm^{-3} of sodium hydroxide, NaOH .

The enthalpy change of neutralisation is the heat evolved when one mole of water is formed during a neutralisation reaction as shown in the equation below.



You will perform a series of experiments by mixing different volumes of **FA 8** and **FA 9** while keeping the total volume of the reaction mixture to be constant. You will determine the temperature change, ΔT , for each reaction and plot a graph of temperature change, ΔT , against volume of **FA 8** used.

The maximum amount of heat is evolved when the acid present is exactly neutralised by the alkali added. You will then analyse the results graphically to determine the equivalence volume of the reaction and hence calculate the value of the enthalpy change of neutralisation, ΔH_{neut} .

For the experiment, you will calculate and record the average initial temperature, T_{avg} , before the reaction occurs when the **FA 8** and **FA 9** are mixed.

Average temperature, T_{avg} , can be obtained by using $T_{\text{avg}} = \frac{T_{\text{FA8}} + T_{\text{FA9}}}{2}$

Temperature change, ΔT , can be obtained by using $\Delta T = T_{\text{max}} - T_{\text{avg}}$

In an appropriate format in the space provided on the next page, prepare a table to record the data for each experiment to an appropriate level of precision:

- all measurements of volumes used,
- T_{max} and the temperature change, ΔT .

(a) Procedure

1. Measure the initial temperature of **FA 8** in the reagent bottle using the thermometer. Record this temperature as T_{FA8} . Wash and dry the thermometer.
2. Repeat step 1 for **FA 9**. Record this temperature as T_{FA9} . Determine T_{avg} using the recorded values of T_{FA8} and T_{FA9} .
3. Place a clean and dry polystyrene cup inside a second polystyrene cup which is placed in a 250 cm³ glass beaker to prevent the cups from tipping over.
4. Using a 50 cm³ measuring cylinder, measure 10.0 cm³ of **FA 8** and transfer the **FA 8** solution into the polystyrene cup.
5. Using a 50 cm³ measuring cylinder, measure 40.0 cm³ of **FA 9** and transfer the **FA 9** solution into the polystyrene cup containing **FA 8**.
6. Using the thermometer, stir the reaction mixture in the polystyrene cup. Measure and record the maximum temperature reached, T_{max} .
7. Discard the contents of the polystyrene cup. Wash and dry the polystyrene cup and the thermometer.

Repeat steps 1 to 7 using 15.0 cm³, 20.0 cm³, 25.0 cm³, 30.0 cm³ and 35.0 cm³ of **FA 8**, each time using an appropriate volume of **FA 9** so that the total volume of each mixture is kept constant.

Results

| V_{FA8} / cm ³ | V_{FA9} / cm ³ | T_{FA8} / °C | T_{FA9} / °C | T_{avg} / °C | T_{max} / °C | ΔT / °C |
|---------------------------------------|---------------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------|
| 10.0 | 40.0 | 30.4 | 30.2 | 30.3 | 36.4 | 6.1 |
| 15.0 | 35.0 | 30.4 | 30.3 | 30.4 | 39.3 | 8.9 |
| 20.0 | 30.0 | 30.2 | 30.2 | 30.2 | 41.7 | 11.5 |
| 25.0 | 25.0 | 30.4 | 30.2 | 30.3 | 41.2 | 10.9 |
| 30.0 | 20.0 | 30.4 | 30.2 | 30.3 | 38.5 | 8.2 |
| 35.0 | 15.0 | 30.4 | 30.4 | 30.4 | 36.2 | 5.8 |

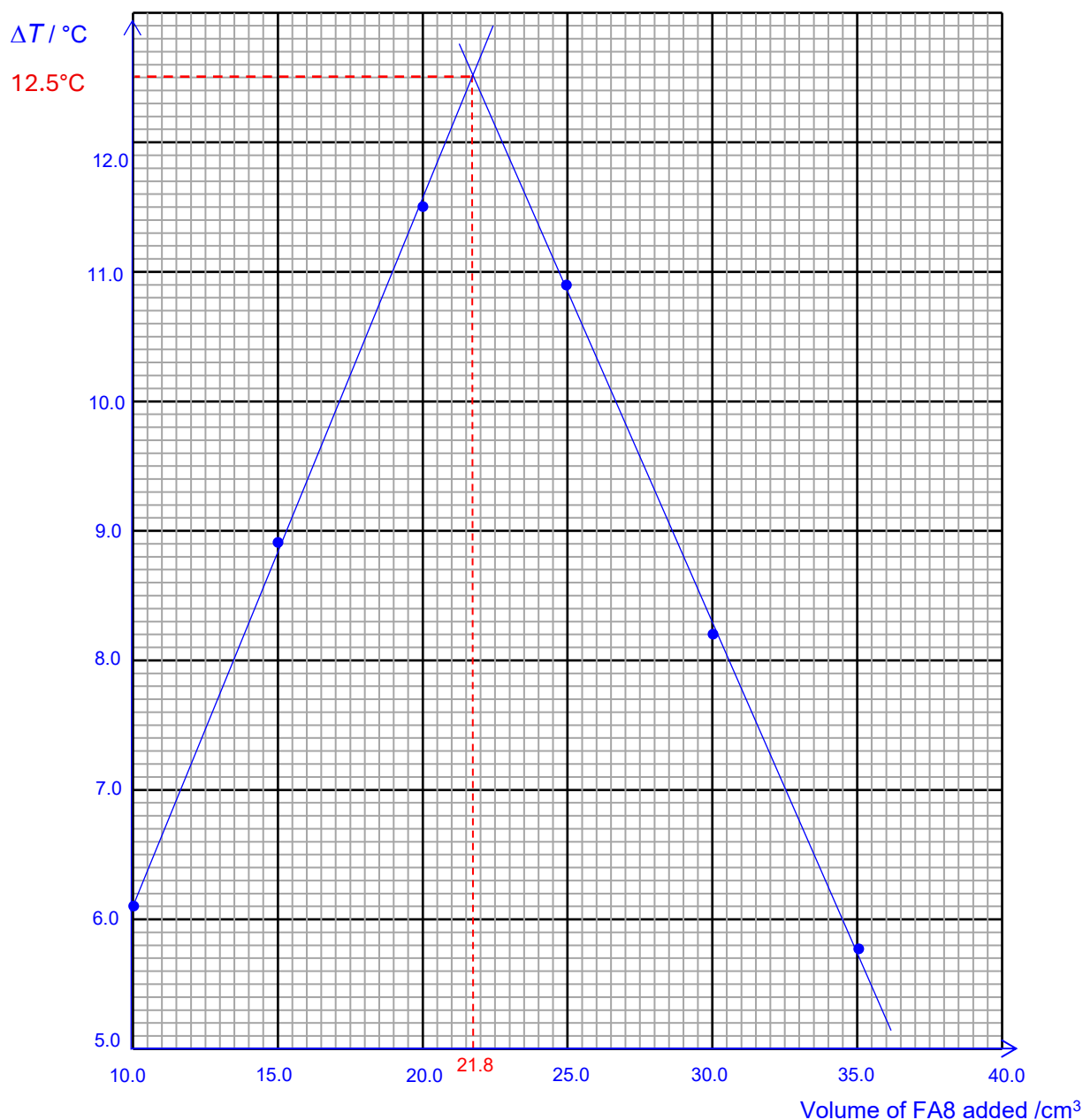
[3]



(b) Using the data obtained, plot a graph of ΔT against volume of **FA 8** added.

Draw two best-fit straight lines for the points plotted, and extrapolate both lines to find

- the temperature change at equivalence point, ΔT_{eq} .
- the volume of **FA 8** added to completely neutralise **FA 9**.



$$\Delta T_{\text{eq}} = \text{.....} \mathbf{+12.5^\circ\text{C}}$$

$$\text{volume of FA 8 required for complete neutralisation} = \text{.....} \mathbf{21.8 \text{ cm}^3} \text{ [5]}$$



- (c) (i) Calculate the volume of **FA 9** required for complete neutralisation. Hence calculate the amount of H_aX in **FA 8** and NaOH in **FA 9** required for complete neutralisation.

$$\text{Volume of FA 9} = 50 - 21.8 = 28.2 \text{ cm}^3$$

$$\text{volume of FA 9 required for complete neutralisation} = 28.2 \text{ cm}^3$$

$$\text{Amount of } H_aX \text{ in FA 8} = 1.00 (21.8/1000) = 0.0218 \text{ mol}$$

$$\text{Amount of NaOH in FA 9} = 1.60 (28.2/1000) = 0.0451 \text{ mol}$$

$$\text{amount of } H_aX \text{ in FA 8} = 0.0218 \text{ mol}$$

$$\text{amount of NaOH in FA 9} = 0.0451 \text{ mol} \quad [2]$$

- (ii) Hence, determine the value of a and deduce a possible identity for H_aX .

Ratio of H_aX : NaOH

$$0.0218 : 0.0451$$

$$\sim 1:2$$

$$\text{Value of } a = 2$$

Since this is a dibasic acid, a possible strong dibasic acid is H_2SO_4 .

$$\text{Value of } a = 2 \quad [1]$$

$$\text{possible identity of } H_aX = H_2SO_4 \quad [1]$$



- (d) Calculate the enthalpy change of neutralisation, H_{neut} , for the reaction between H_aX and NaOH .

Assume that the specific heat capacity of the reaction mixture is $4.18 \text{ J g}^{-1} \text{ K}^{-1}$ and its density is 1.00 g cm^{-3} .

$$\begin{aligned} Q &= mc\Delta T \\ &= 50(4.18)(+12.5) \\ &= 2612.5 \text{ J} \end{aligned}$$

$$\begin{aligned} \Delta H_{\text{neut}} &= -2612.5 / 0.0451 \\ &= -57927 \text{ J mol}^{-1} \\ &= \underline{-57.9 \text{ kJ mol}^{-1}} \end{aligned}$$

$$\Delta H_{\text{neut}} = \underline{-57.9 \text{ kJ mol}^{-1}} \dots\dots\dots [2]$$

- (e) In another experiment, **FA 9** was replaced by a solution of methylamine, CH_3NH_2 , a weak monoacidic base of equal concentration. State and explain the effect on the following.

- ΔT_{eq}

ΔT_{eq} will be **decreased/lowered**. Methylamine is a **weak base** and does not dissociate completely in aqueous solution. **Some of the heat evolved from the neutralisation reaction would be absorbed to allow for complete dissociation of methylamine** to reach the equivalence point.

- volume of **FA 8** required

Volume of **FA 8** **does not change** . **Exactly equal amounts of the acid neutralised by the alkali/ reacts based on stoichiometric ratio** at the equivalence point as the concentrations of the acid and the alkali are the same

..... [3]

[Total: 17]

4 Planning

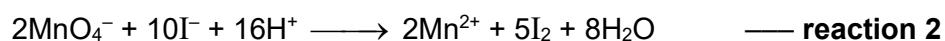
When potassium manganate(VII) reacts with sodium ethanedioate and sulfuric acid, a redox reaction occurs as shown below:



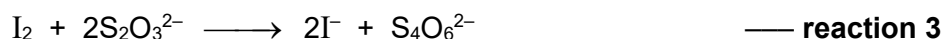
A product that is generated during the course of the reaction and helps to speed up the reaction rate is known as an autocatalyst. The autocatalyst for the above reaction is Mn^{2+} .

The kinetics of this reaction can be investigated by determining the concentration of MnO_4^- over the course of the reaction in a continuous monitoring experiment. Fixed aliquots (portions) of the reaction mixture are withdrawn at regular time intervals and added to an excess of potassium iodide.

The MnO_4^- in the aliquots reacts with excess iodide as shown below.



The amount of iodine formed can then be determined by titration with the sodium thiosulfate solution as shown below.



The volume of sodium thiosulfate used can then be used to determine the concentration of MnO_4^- .

- (a) In reaction 1, both sodium ethanedioate and sulfuric acid are used in large excess. Explain the purpose of using a large excess of these solutions.

This is to ensure that the concentration of sodium ethanedioate and sulfuric acid remains almost constant throughout the reaction / concentration do not affect rate.

..... [1]

- (b) Using the information given above, you are required to write a plan to investigate the kinetics of the autocatalytic experiment via the determination of the concentration of MnO_4^- at regular timed intervals.

You may assume that you are provided with the following:

- 25 cm³ of 0.0500 mol dm⁻³ potassium manganate(VII), KMnO_4
- 0.500 mol dm⁻³ sodium ethanedioate, $\text{Na}_2\text{C}_2\text{O}_4$
- 1.00 mol dm⁻³ sulfuric acid, H_2SO_4
- 0.200 mol dm⁻³ potassium iodide, KI
- **M** mol dm⁻³ $\text{Na}_2\text{S}_2\text{O}_3$
- starch indicator
- stopwatch
- apparatus and chemicals normally found in a school or college laboratory.



Your plan should include:

- using the 25.0 cm³ of KMnO₄ provided, justification for the volume of the remaining reactants to be used in **reaction 1**, so that a minimum of **6 aliquots of 10 cm³** of the reaction mixture can be withdrawn
- determining a suitable concentration for the sodium thiosulfate, Na₂S₂O₃, to be used
- details of the apparatus you would use
- details of the experimental procedure, in particular the preparation of the reaction mixture and the titration with Na₂S₂O₃
- an outline of the results to be recorded and a sketch of the expected graph you would obtain

[8]

Suggested Mark Scheme

Pre-calculation

Assuming 10 cm³ aliquot is withdrawn for each titration, minimum volume required for reaction 1 is 60 cm³.

Volume of MnO₄⁻ used = 25.0 cm³

Volume of Na₂C₂O₄ used = 50.0 cm³

Volume of H₂SO₄ used = 25.0 cm³

Total volume used for reaction 1 = 100 cm³

Calculation for concentration of Na₂S₂O₃ to be used (assuming that MnO₄⁻ is the limiting reagent)

$$\begin{aligned}\text{Amount of MnO}_4^- \text{ present in reaction 1} &= 0.0500 \times \frac{25.0}{1000} \\ &= 1.25 \times 10^{-3} \text{ mol}\end{aligned}$$

$$\begin{aligned}\text{Amount of MnO}_4^- \text{ present in 10 cm}^3 \text{ aliquot} &= 1.25 \times 10^{-3} \times \frac{10.0}{100} \\ &= 1.25 \times 10^{-4} \text{ mol}\end{aligned}$$

$$\begin{aligned}\text{Amount of I}_2 \text{ produced in reaction 2} &= 1.25 \times 10^{-4} \times \frac{5}{2} \\ &= 3.13 \times 10^{-4} \text{ mol}\end{aligned}$$

$$\begin{aligned}\text{Amount of S}_2\text{O}_3^{2-} \text{ required in reaction 3} &= 3.125 \times 10^{-4} \times 2 \\ &= 6.25 \times 10^{-4} \text{ mol}\end{aligned}$$

Assuming a titre volume of 25.00 cm³ (acceptable range 10–30 cm³)

$$\begin{aligned}[\text{S}_2\text{O}_3^{2-}] \text{ used for titration} &= 6.25 \times 10^{-4} \div \frac{25.00}{1000} \\ &= 0.0250 \text{ mol dm}^{-3}\end{aligned}$$



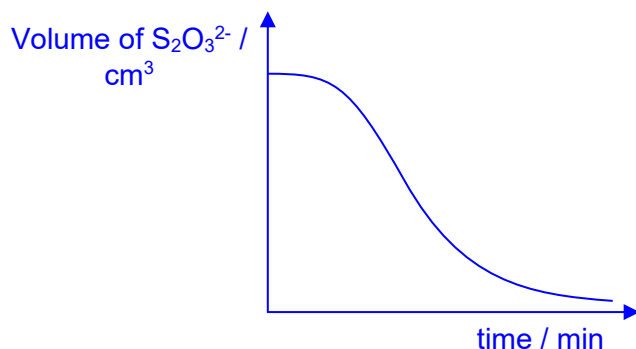
Preparation of reaction mixture

1. Using a **100 cm³ measuring cylinder**, measure 50.0 cm³ of Na₂C₂O₄ and 25.0 cm³ H₂SO₄ into a 250 cm³ conical flask.
2. Using a **50 cm³ measuring cylinder**, measure 25.0 cm³ of KMnO₄.
3. Pour the MnO₄⁻ to the reaction mixture in the conical flask and **start the stopwatch immediately. Ensure thorough mixing by swirling the flask.**

Titration with sodium thiosulfate

4. Using a **10 cm³ measuring cylinder**, measure 10.0 cm³ of KI and add it into a separate 100 cm³ conical flask.
5. At about 1 (or 2) min, **pipette 10.0 cm³ of the reaction mixture** into the separate conical flask containing the 10.0 cm³ of KI solution. Label conical flask 1.
6. Fill a 50.00 cm³ **burette** with Na₂S₂O₃.
7. Titrate the iodine in conical flask 1 against Na₂S₂O₃.
8. When the colour of the solution turns pale yellow, add about **1 cm³ of starch indicator.** The solution will turn **blue-black.**
9. Continue the titration and the end-point is reached when the blue-black **colour just disappears.**
10. Repeat steps 4 – 9 five more times at **2 – 4 minute intervals**, transferring the aliquots into dry and clean conical flasks.
11. Record down the titre volume against time for all 6 experiments.

Shape of graph expected



[Total: 9]



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, $\text{Cl}^-(\text{aq})$ | 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 |

