



VICTORIA JUNIOR COLLEGE
 JC2 PRELIMINARY EXAMINATIONS
 Higher 2

CANDIDATE
 NAME

CT GROUP

CHEMISTRY

9729//04

Paper 4 Practical

30 August 2021

2 hours 30 minutes

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name and CT group 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 a HB pencil for any diagrams or graphs.
 Do not use staples, paper clips, glue or correction fluid.

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

The 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 the last page.

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.

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| Shift | |
| | |
| Laboratory | |
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| For Examiner's Use | |
| 1 | |
| 2 | |
| 3 | |
| 4 | |
| Total | |

This document consists of **19** printed pages (excluding the QA notes).

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

1 Determination the extent of oxidation of iron(II) solution exposed to air

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.

It is essential to dissolve solid samples of iron(II) compounds in acids such as dilute sulfuric acid instead of water. This is to prevent excessive oxidation of iron(II) ions to iron(III) ions in the presence of oxygen over a period of time.

FA 1 is $0.020 \text{ mol dm}^{-3}$ acidified potassium manganate(VII), KMnO_4 .

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

FA 3 is iron(II) salt dissolved in water and left standing for an extended period of time.

Using the apparatus and chemicals provided, you are required to determine the extent of oxidation of iron(II) salt in **FA 3**.

(a) Dilution of FA 3

1. Pipette 25.0 cm^3 of **FA 3** into a 100 cm^3 volumetric flask.
2. Make up to the contents of the flask to the 100 cm^3 mark with deionised water. Shake to obtain a homogeneous solution.
3. Label this solution **diluted FA 3** which is to be used for (b) and (c).

(b) Determination of amount of iron(II) ions originally present in FA 3.

1. Add 50 cm^3 of **diluted FA 3** using a measuring cylinder into a 100 cm^3 beaker.
2. Add about 2 spatula of zinc powder into the beaker and stir.
3. Filter the mixture into a boiling tube.
4. Pipette 10.0 cm^3 of the filtrate into a conical flask.
5. Add 10 cm^3 of **FA 2** into the conical flask using a measuring cylinder.
6. Titrate this solution against **FA 1** from a burette until the first **permanent** pale pink colour is obtained.
7. Record your titration results, to an appropriate level of precision, in the space below.

You are to perform this titration only once.

Results:

[2]

(c) Determination of amount of iron(II) remaining in FA 3.

1. Pipette 10.0 cm³ of **diluted FA 3** into a conical flask.
2. Add 10 cm³ of **FA 2** into the conical flask using a measuring cylinder.
3. Titrate this solution against **FA 1** from a burette until the first **permanent** pale pink colour is obtained.
4. Record your titration results, to an appropriate level of precision, in the space below.
5. Repeat the titration as many times as necessary to obtain consistent results.

Results:

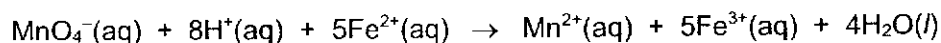
[3]

(d) Calculations

- (i) From your titration results in (c), calculate the average volume of **FA 1** used.

[1]

- (ii) Calculate the amount of iron(II) in 10.0 cm³ of **diluted FA 3**.



amount of iron(II) in **diluted FA 3** =

[1]

- (iii) Calculate the amount iron(II) in 25.0 cm³ of FA 3.

amount of iron(II) in FA 3 =
[1]

- (iv) Using your titration results in (b), calculate the amount iron(II) originally present in 25.0 cm³ of FA 3.

amount of iron(II) originally present in FA 3 =
[1]

- (v) Calculate the percentage of iron(II) in FA 3 oxidised upon standing for an extended period of time.

percentage of iron(II) in FA 3 oxidised =
[2]

(e) Evaluations

- (i) State one assumption made for the addition of zinc powder in (b). Explain how the titre volume would be affected if the assumption is **not** valid.

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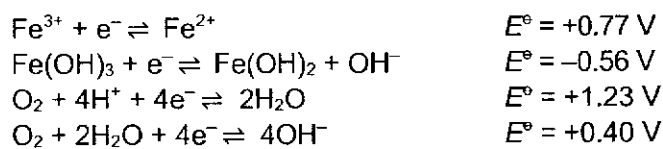
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[2]

- (ii) With the help of relevant calculations, show that the iron(II) is more stable in air under acidic conditions compared to alkaline conditions.



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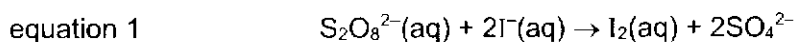
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[2]

[Total: 15]

2 Determination of the activation energy of the reaction between ammonium peroxodisulfate and potassium iodide.

Equation 1 represents the reaction between peroxodisulfate ions and iodide ions.

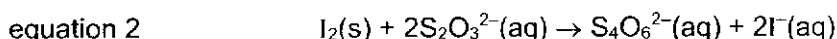


The reaction is first order with respect to both the concentration of peroxodisulfate ions and the concentration of iodide ions.

$$\text{rate} = k[\text{S}_2\text{O}_8^{2-}][\text{I}^-] \quad \text{where } k = \text{rate constant}$$

When starch is added to the reaction mixture, a blue colour is immediately seen due to the formation of an iodine–starch complex.

If a small amount of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, is also present in the reaction mixture, the formation of the blue colour is delayed. The $\text{Na}_2\text{S}_2\text{O}_3$ reacts with I_2 as shown in equation 2.



The activation energy, E_a , for the reaction can be obtained using the Arrhenius Equation:

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

where k is the rate constant,

A is the pre-exponential constant

R is molar gas constant and

T is the reaction temperature in Kelvin,

The activation energy, E_a , of the reaction can be determined by performing a series of experiments where the reaction mixture is kept constant, but different reaction temperatures are used, and the same end-point is timed. Plotting $\ln k$ against $1/T_K$ will give a best-fit straight line where the gradient of the line is $-E_a/R$.

FA 4 is $0.200 \text{ mol dm}^{-3}$ potassium iodide, KI.

FA 5 is $0.00500 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 6 is $0.100 \text{ mol dm}^{-3}$ ammonium peroxodisulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$.

Solution S is starch solution.

You are to perform a series of five experiments, at different temperatures, in which the concentrations of **FA 4**, **FA 5** and **FA 6** are kept constant. The time taken for the dark blue colour to form allows the reaction rate to be determined.

(a) Procedure

You will attempt **five** experiments. In each experiment, **solution 1** will be prepared as described below and a standard volume of **FA 6** will then be added.

For each experiment you will:

- note the time in seconds, t , taken for the solution to turn dark blue
- note the values of the initial, T_i , and final, T_f , temperatures

You will then calculate values for

- T_{avg} , the average temperature, of the T_i and T_f values
- T_K , the average temperature in Kelvin ($0\text{ }^\circ\text{C} = 273\text{ K}$)
- $1/t$
- $\ln(1/t)$
- $1/T_K$

Prepare a table in the space provided on the next page in which to record to an appropriate level of precision:

- all values of T and t
- all the values of $1/t$, $\ln(1/t)$ and $1/T_K$

For each experiment, prepare **solution 1** as follows.

Using appropriate measuring cylinders, transfer to a dry 100 cm^3 beaker:

- 20.0 cm^3 of **FA 4**
- 10.0 cm^3 of **FA 5**
- 5.0 cm^3 of **solution S**

Then stir the contents of the beaker using a glass rod.

(i) Experiment 1

The end-point of the reaction is the first appearance of a blue colour.

Note: Insufficient swirling of the reaction mixture may lead to a blue colour appearing before the true end-point is reached.

1. Place the beaker containing **solution 1** on a white tile.
2. Using an appropriate measuring cylinder, transfer 20.0 cm^3 of **FA 6** to the beaker. Start the stopwatch during this addition.
3. Mix the contents in the beaker by **thoroughly** stirring with a glass rod.
4. Using a thermometer, note the initial temperature, T_i , of the reaction mixture.
5. Stop the stopwatch when the solution **first** turns blue.
6. Note the final temperature, T_f , of the reaction mixture.
7. Record the time taken, t , to the nearest second.
8. Wash the beaker and thermometer thoroughly with water and dry them.

(ii) Experiment 2

9. Prepare **solution 1** as described earlier.
10. Half-fill a 250 cm^3 beaker with ice/water to make a cooling bath.
11. Place the 100 cm^3 beaker, containing **solution 1**, into the cooling bath and carefully stir **solution 1** until its temperature is between $5\text{ }^\circ\text{C}$ and $10\text{ }^\circ\text{C}$.
12. Remove the beaker from the cooling bath and repeat steps 2 – 8.

You have now performed the slowest experiment.

(iii) Experiment 3

13. Prepare **solution 1** as described earlier.
14. Half-fill a 250 cm³ beaker with hot water to make a heating bath.
15. Place the 100 cm³ beaker, containing **solution 1**, into the heating bath and carefully stir **solution 1** until its temperature is between 50 °C and 55 °C.
16. Remove the beaker from the heating bath and repeat steps 2 – 8.

You have now performed the fastest experiment.

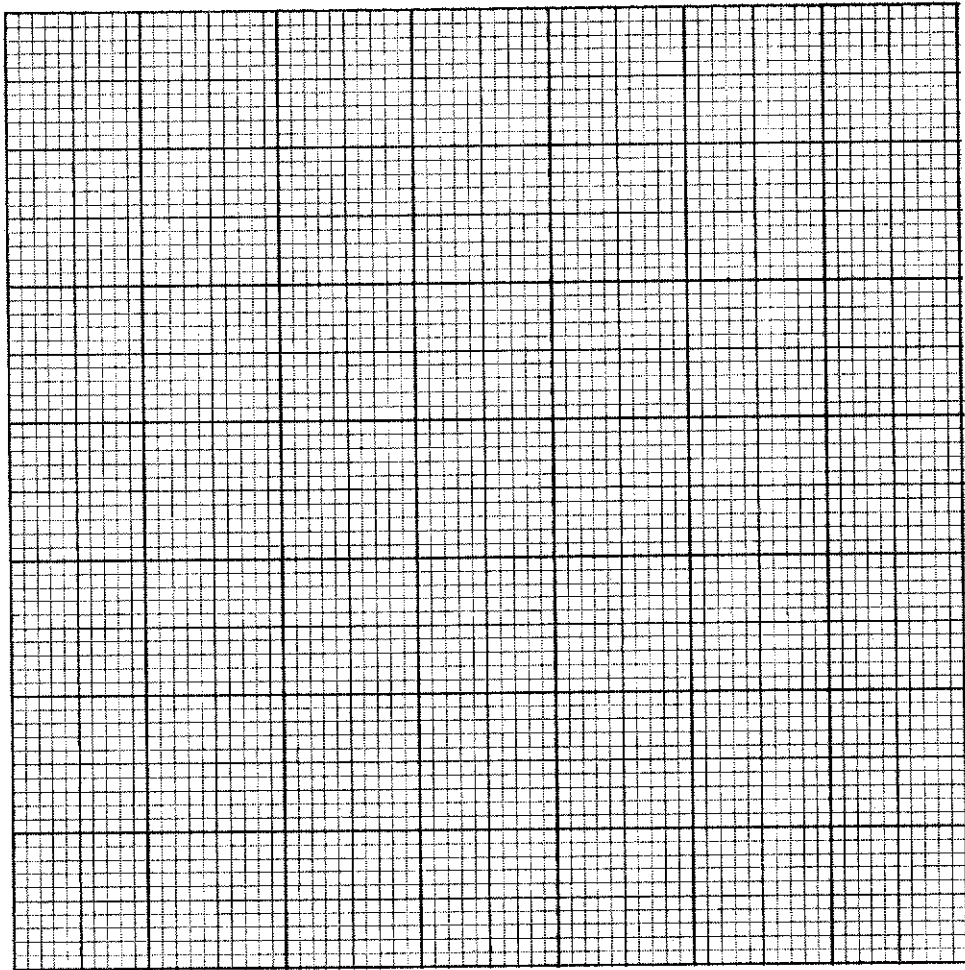
(iv) Experiments 4 and 5

Select **two** other suitable reaction temperatures, between the temperatures of experiments 2 and 3, for use in two additional experiments. In each case, use the heating bath or cooling bath to bring **solution 1** to an appropriate temperature and perform these experiments.

Results

[4]

- (b) (i) Plot a graph of $\ln(1/t)$ on the y-axis against $1/T_K$ on the x-axis. Draw the best-fit straight-line taking into account all of your plotted points.



[3]

- (ii) Calculate the gradient of the line, showing clearly how you did this.

gradient = [1]

- (iii) Determine a value for the activation energy, E_a . Show your working.
[$R = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$]

activation energy, $E_a = \dots\dots\dots$ [1]

- (c) This version of the 'Iodine Clock' experiment is very reliable. With care, it is possible to perform a reaction that will change colour after a specified time interval.

Using **your graph**, calculate the temperature, in °C, of the reaction mixture that would result in the reaction mixture turning blue after **58 seconds**. Show your working.

temperature required =
[3]

- (d) Another student performed **experiment 1**. She repeated the experiment but, before adding the **FA 6** solution, she added a few drops of **FA 3** (from Question 1) to **solution 1**. She found that this addition considerably reduced the time required for a dark blue colour to form.

With the aid of relevant equations, explain the mode of action **FA 3** in the reaction between peroxodisulfate and iodide ions.

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[2]

[Total: 14]

3 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 3.1** and **3.2**.

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) Inorganic analysis

You are provided with the solutions, **FA 7** and **FA 8**.

FA 7 is a solution containing one cation and one anion. The cation is singly charged and is **not** listed in the Qualitative Analysis notes.

FA 8 is prepared in the following way:

A metal oxide, M_2O , is added in excess to **FA 7**. A disproportionation reaction occurs, with a red-brown solid formed as one of the products. The red-brown solid, together with excess M_2O , is then removed via filtration. The filtrate collected is **FA 8**.

Table 3.1

| | tests | observations |
|-------|--|--------------|
| (i) | Add 1 cm depth of FA 7 in a test-tube, followed by 1 cm depth of aqueous sodium carbonate. | |
| (ii) | Add 1 cm depth of FA 7 in a test-tube. Add aqueous barium chloride, followed by aqueous hydrochloric acid. | |
| (iii) | Add 1 cm depth of FA 8 to a test-tube. Add aqueous ammonia slowly, with shaking, until no further change is seen. Then add FA 7 slowly, with shaking, until no further change is seen. | |

| | | |
|------|--|--|
| (iv) | Add 1 cm depth of FA 8 in a boiling tube, followed by 1 cm depth of aqueous sodium carbonate. Heat the mixture for about half a minute. You do not need to test for any gas. | |
|------|--|--|

[4]

(b) (i) Suggest the identity of the following ions:

Cation present in **FA 7**:

Anion present in **FA 7**:

Cation present in **FA 8**:

[2]

(ii) Hence, construct the full equation for the reaction between M_2O and **FA 7** to form **FA 8**.

.....
[1]

(iii) When aqueous sodium carbonate was added to **FA 8** in (a)(iv), compound **R** is formed as one of the products. Compound **R** contains the cation of **FA 8** and two anions, **S** and **T**. The cation, **S** and **T** in compound **R** are present in the molar ratio of 2 : 2 : 1 respectively.

By considering the ions originally present in aqueous sodium carbonate and your answer in (b)(i), suggest the formula of the product **R**.

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[1]

(iv) Two gases were evolved during heating in (a)(iv) but it is difficult for you to detect them due to the short duration of the heating process. One of the gases evolved was water vapour.

Suggest the identity of the other gas evolved. Hence, construct an equation for the reaction that took place during heating in (a)(iv).

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[1]

(c) Organic analysis

In this question, you will deduce the structure of an organic compound, **FA 9**.

FA 9 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 3.2

| | tests | observations |
|------|--|--------------|
| (i) | <p>Place about 2 cm depth of aqueous sulfuric acid in a test-tube.</p> <p>To this test-tube, add 15 drops of FA 9, followed by 1 drop of aqueous potassium manganate(VII).</p> <p>Warm the mixture in the hot water bath for two minutes.</p> | |
| (ii) | <p>Add 1 cm depth of aqueous silver nitrate to a test-tube.</p> <p>Then slowly add 1 cm depth of aqueous sodium hydroxide.</p> <p>Add aqueous ammonia slowly, with shaking, until the precipitate just dissolves. You may use a clean glass rod to stir the mixture and help dissolve the precipitate.</p> <p>To this mixture, add 10 drops of FA 9.</p> <p>Place 50 cm³ of hot water in a beaker and note its temperature. If the temperature is higher than 60 °C, add a small amount of water at room temperature, stir and note the temperature.</p> <p>Keep adding small amounts of water until the temperature of the water is 60 °C or lower.</p> <p>Place the test-tube containing the mixture in the water bath for one minute.</p> | |

| | | |
|-------|---|---|
| (iii) | Place about 1 cm depth of FA 9 in a test-tube. To this test-tube, add 2,4-dinitrophenylhydrazine dropwise. | orange ppt. formed |
| (iv) | Place about 15 drops of FA 9 and add 10 drops of aqueous sodium hydroxide in a test-tube. Now add iodine solution dropwise, until a permanent orange/red colour is obtained. Warm the mixture in the hot water bath for two minutes. | |
| (v) | Place 1 cm depth of FA 9 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]

- (d) (i) Observations from (c)(iii), together with those of either (c)(i) or (c)(ii), can be used to identify one of the functional groups present in **FA 9**.

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

Functional group:

Explanation:

.....

[1]

- (ii) Using observations from **either (c)(iv) or (v)**, identify the other functional group present in **FA 9**.

Quote evidence from the relevant test to support your conclusion.

Functional group:

Evidence:

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[1]

- (iii) Suggest two possible structures of **FA 9** that are consistent with all the observations in **Table 3.2**.

[2]

[Total: 15]

4 Planning

FA 10 is a solution of aqueous potassium hydroxide, KOH, with a rough concentration of 0.2 mol dm^{-3} . **FA 11** is a mixture of $0.100 \text{ mol dm}^{-3}$ aqueous sulfuric acid, H_2SO_4 , and $0.100 \text{ mol dm}^{-3}$ sodium sulfate, Na_2SO_4 .

To determine the accurate concentration of potassium hydroxide in **FA 10**, a series of experiments can be conducted in which the temperature change in each experiment is recorded. In each experiment, a different volume of **FA 11** is used but the volume of **FA 10** is kept constant. The total volume of the reaction mixture is also kept constant by adding an appropriate volume of water.

A graph of *temperature change* against the *volume of acid added* is then plotted. In this graph, two best-fit lines are drawn and extrapolated until they cross one another. The point of intersection is the equivalence point of the chemical reaction.

With the equivalence point determined from the graph, the accurate concentration of aqueous potassium hydroxide can then be determined.

- (a) Using the information given above, write an experimental plan to determine the equivalence point for the reaction between **FA 10** and **FA 11**.

You may assume that you are provided with:

- **FA 10**, which is a solution of KOH with a rough concentration 0.2 mol dm^{-3} ,
- **FA 11**, which is a mixture of $0.100 \text{ mol dm}^{-3}$ H_2SO_4 and $0.100 \text{ mol dm}^{-3}$ Na_2SO_4 ,
- the equipment normally found in a school or college laboratory.

In your plan, you should include brief details of:

- calculation of the approximate volume of **FA 11** required to completely neutralise a fixed volume of 20.0 cm^3 of **FA 10**,
- a table containing the volumes of each reagent to be added for six experiments,
- the apparatus you would use,
- the procedure you would follow,
- the measurements you would make to allow a graph of *temperature change* against the *volume of acid added* to be drawn.

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Dotted lines for writing.

[5]

- (b) (i) Sketch on **Figure 4.1** the graph you would expect to get from your results.

Indicate on the graph the equivalence point, **V** (in cm^3) and the maximum temperature rise, **A**.

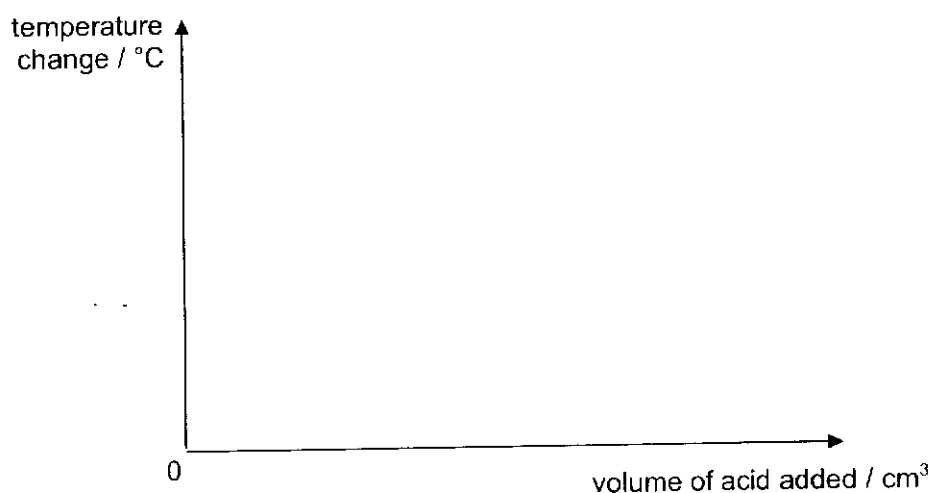


Figure 4.1

[1]

- (ii) Show how the data obtained from the graph in (b)(i) can be used to determine
- the accurate concentration of potassium hydroxide, and
 - enthalpy change of neutralisation, ΔH_n in J mol^{-1} , assuming x J is required to raise the temperature of 1 cm^3 of solution by $1 \text{ }^\circ\text{C}$ and the total volume of the mixture is $V_t \text{ cm}^3$

[2]

(c) In another experiment, you are given **FA 12**, which is a mixture of $0.100 \text{ mol dm}^{-3}$ aqueous ethanedioic acid, $\text{HO}_2\text{CCO}_2\text{H}$ and $0.1000 \text{ mol dm}^{-3}$ sodium ethanedioate, $\text{NaO}_2\text{CCO}_2\text{Na}$.

(i) Sketch on **Figure 4.1** another graph to show the results you would expect to obtain when **FA 12** is used instead of **FA 11**. Label the graph with (c)(i).

[1]

(ii) Explain the difference in the two graphs in **Figure 4.1**.

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[2]

[Total: 11]

Victoria Junior College
2021 H2 Chemistry Prelim Exam 9729/4
Suggested Answers

1 Determination the extent of oxidation of iron(II) solution exposed to air

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.

It is essential to dissolve solid samples of iron(II) compounds in acids such as dilute sulfuric acid instead of water. This is to prevent excessive oxidation of iron(II) ions to iron(III) ions in the presence of oxygen over a period of time.

FA 1 is $0.020 \text{ mol dm}^{-3}$ acidified potassium manganate(VII), KMnO_4 .

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

FA 3 is iron(II) salt dissolved in water and left standing for an extended period of time.

Using the apparatus and chemicals provided, you are required to determine the extent of oxidation of iron(II) salt in **FA 3**.

(a) Dilution of FA 3

1. Pipette 25.0 cm^3 of **FA 3** into a 100 cm^3 volumetric flask.
2. Make up to the contents of the flask to the 100 cm^3 mark with deionised water. Shake to obtain a homogeneous solution.
3. Label this solution **diluted FA 3** which is to be used for **(b)** and **(c)**.

(b) Determination of amount of iron(II) ions originally present in FA 3.

1. Add 50 cm^3 of **diluted FA 3** using a measuring cylinder into a 100 cm^3 beaker.
2. Add about 2 spatula of zinc powder into the beaker and stir.
3. Filter the mixture into a boiling tube.
4. Pipette 10.0 cm^3 of the filtrate into a conical flask.
5. Add 10 cm^3 of **FA 2** into the conical flask using a measuring cylinder.
6. Titrate this solution against **FA 1** from a burette until the first **permanent** pale pink colour is obtained.
7. Record your titration results, to an appropriate level of precision, in the space below.

You are to perform this titration only once.

Results:

| | |
|---|--------------|
| Experiment | 1 |
| Final burette reading / cm^3 | 19.00 |
| Initial burette reading / cm^3 | 0.00 |
| Volume of FA 1 used / cm^3 | 19.00 |

For both **(b)** and **(c)** table
Correct headers with units;
Tabulation of ALL burette readings to 2.d.p.

[2]

(c) Determination of amount of iron(II) remaining in FA 3.

1. Pipette 10.0 cm³ of **diluted FA 3** into a conical flask.
2. Add 10 cm³ of **FA 2** into the conical flask using a measuring cylinder.
3. Titrate this solution against **FA 1** from a burette until the first **permanent** pale pink colour is obtained.
4. Record your titration results, to an appropriate level of precision, in the space below.
5. Repeat the titration as many times as necessary to obtain consistent results.

Results:

| Experiment | 1 | 2 |
|---|-------|-------|
| Final burette reading / cm ³ | 13.00 | 13.00 |
| Initial burette reading / cm ³ | 0.00 | 0.00 |
| Volume of FA 1 used / cm ³ | 13.00 | 13.00 |

At least two consistent readings ± 0.10 cm³
Accuracy [2m]

[3]

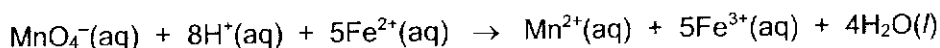
(d) Calculations

- (i) From your titration results in (c), calculate the average volume of
- FA 1**
- used.

$$\begin{aligned} \text{Average volume of FA 1 used} &= (13.00 + 13.00) \div 2 \\ &= 13.00 \text{ cm}^3 \text{ (2 d.p.)} \end{aligned}$$

[1]

- (ii) Calculate the amount of iron(II) in 10.0 cm
- ³
- of
- diluted FA 3**
- .



$$\begin{aligned} \text{Amount of MnO}_4^- \text{ required} \\ &= 13.00/1000 \times 0.020 \\ &= 2.60 \times 10^{-4} \text{ mol (3 s.f.)} \end{aligned}$$

$$\begin{aligned} \text{Amount of iron(II) in 10.0 cm}^3 \text{ of diluted FA 3} \\ &= 2.60 \times 10^{-4} \times 5 \\ &= 1.30 \times 10^{-3} \text{ mol (3 s.f.)} \end{aligned}$$

$$\text{Amount of iron(II) in diluted FA 3} = 1.30 \times 10^{-3} \text{ mol} \quad [1]$$

- (iii) Calculate the amount iron(II) in 25.0 cm
- ³
- of
- FA 3**
- .

$$\begin{aligned} \text{Amount of iron(II) in 100 cm}^3 \text{ of diluted FA 3} \\ &= 1.30 \times 10^{-3} \times \frac{100}{10.0} \\ &= 1.30 \times 10^{-2} \text{ mol} \end{aligned}$$

Number of moles before dilution = Number of moles after dilution

$$\begin{aligned} \text{Amount of iron(II) in 25.0 of FA 3} \\ &= 1.30 \times 10^{-2} \text{ mol (3 s.f.)} \end{aligned}$$

$$\text{Amount of iron(II) in FA 3} = 1.30 \times 10^{-2} \text{ mol}$$

[1]

- (iv) Using your titration results in (b), calculate the amount iron(II) originally present in 25.0 cm³ of FA 3.

$$\begin{aligned} \text{Amount of MnO}_4^- \text{ required} \\ &= 19.00/1000 \times 0.020 \\ &= 3.80 \times 10^{-4} \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Amount of iron(II) originally present in 10.0 cm}^3 \text{ of diluted FA 3} \\ &= 3.80 \times 10^{-4} \times 5 \\ &= 1.90 \times 10^{-3} \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Amount of iron(II) in 25.0 of FA 3} \\ &= \text{Amount of iron(II) originally present in 100 cm}^3 \text{ of diluted FA 3} \\ &= 1.90 \times 10^{-3} \times \frac{100}{10.0} \\ &= 1.90 \times 10^{-2} \text{ mol (3 s.f.)} \end{aligned}$$

$$\text{Amount of iron(II) originally present in FA 3} = 1.90 \times 10^{-2} \text{ mol} \quad [1]$$

- (v) Calculate the percentage of iron(II) in FA 3 oxidised upon standing for an extended period of time.

$$\begin{aligned} \text{Amount of iron(II) oxidised} \\ &= 1.90 \times 10^{-2} - 1.30 \times 10^{-2} \\ &= 6.00 \times 10^{-3} \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Percentage of iron(II) oxidised} \\ &= \frac{6.00 \times 10^{-3}}{1.90 \times 10^{-2}} \times 100 \% \\ &= 31.6 \% \text{ (3 s.f.)} \end{aligned}$$

$$\text{Percentage of iron(II) in FA 3 oxidised} = 31.6 \% \quad [2]$$

(e) Evaluations

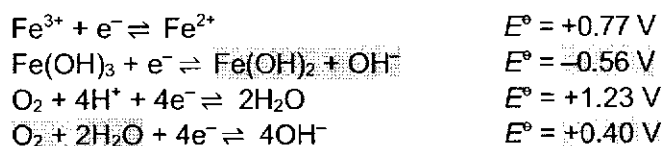
- (i) State one assumption made for the addition of zinc powder in (b). Explain how the titre volume would be affected if the assumption is **not** valid.

The assumption was that all of the Fe³⁺ was reduced to Fe²⁺ by the zinc powder.

If some Fe³⁺ remained, less FA 1 will be needed to oxidise the Fe²⁺ present. Thus, titre volume in (b) would be lower.

[2]

- (ii) With the help of relevant calculations, show that the iron(II) is more stable in air under acidic conditions compared to alkaline conditions.



In acidic conditions,

$$\begin{aligned} E^{\circ}_{\text{cell}} &= E^{\circ}_{\text{red}} - E^{\circ}_{\text{ox}} \\ &= +1.23 - (+0.77) \\ &= 0.46 \text{ V} \end{aligned}$$

In alkaline conditions,

$$\begin{aligned} E^{\circ}_{\text{cell}} &= E^{\circ}_{\text{red}} - E^{\circ}_{\text{ox}} \\ &= +0.40 - (-0.56) \\ &= 0.96 \text{ V} \end{aligned}$$

for correct calculation of E°_{cell} values.

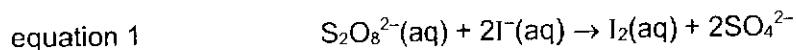
Since E°_{cell} in acidic medium is less positive than that in alkaline medium, iron(II) undergoes oxidation less readily in acidic conditions i.e. iron(II) is more stable in acidic medium.

[2]

[Total: 15]

2 Determination of the activation energy of the reaction between ammonium peroxydisulfate and potassium iodide.

Equation 1 represents the reaction between peroxydisulfate ions and iodide ions.

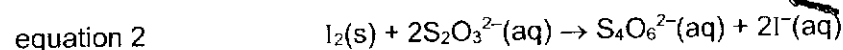


The reaction is first order with respect to both the concentration of peroxydisulfate ions and the concentration of iodide ions.

$$\text{rate} = k[\text{S}_2\text{O}_8^{2-}][\text{I}^{-}] \quad \text{where } k = \text{rate constant}$$

When starch is added to the reaction mixture, a blue colour is immediately seen due to the formation of an iodine–starch complex.

If a small amount of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, is also present in the reaction mixture, the formation of the blue colour is delayed. The $\text{Na}_2\text{S}_2\text{O}_3$ reacts with I_2 as shown in equation 2.



The activation energy, E_a , for the reaction can be obtained using the Arrhenius Equation:

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

where k is the rate constant,
 A is the pre-exponential constant
 R is molar gas constant and
 T is the reaction temperature in Kelvin,

The activation energy, E_a , of the reaction can be determined by performing a series of experiments where the reaction mixture is kept constant, but different reaction temperatures are used, and the same end-point is timed. Plotting $\ln k$ against $1/T_K$ will give a best-fit straight line where the gradient of the line is $-E_a/R$.

FA 4 is $0.200 \text{ mol dm}^{-3}$ potassium iodide, KI.

FA 5 is $0.00500 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 6 is $0.100 \text{ mol dm}^{-3}$ ammonium peroxodisulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$.

Solution S is starch solution.

You are to perform a series of five experiments, at different temperatures, in which the concentrations of **FA 4**, **FA 5** and **FA 6** are kept constant. The time taken for the dark blue colour to form allows the reaction rate to be determined.

(a) Procedure

You will attempt **five** experiments. In each experiment, **solution 1** will be prepared as described below and a standard volume of **FA 6** will then be added.

For each experiment you will:

- note the time in seconds, t , taken for the solution to turn dark blue
- note the values of the initial, T_i , and final, T_f , temperatures

You will then calculate values for

- T_{avg} , the average temperature, of the T_i and T_f values
- T_K , the average temperature in Kelvin ($0^\circ\text{C} = 273 \text{ K}$)
- $1/t$
- $\ln(1/t)$
- $1/T_K$

Prepare a table in the space provided on the next page in which to record to an appropriate level of precision:

- all values of T and t
- all the values of $1/t$, $\ln(1/t)$ and $1/T_K$

For each experiment, prepare **solution 1** as follows.

Using appropriate measuring cylinders, transfer to a dry 100 cm^3 beaker:

- 20.0 cm^3 of **FA 4**
- 10.0 cm^3 of **FA 5**
- 5.0 cm^3 of **solution S**

Then stir the contents of the beaker using a glass rod.

(i) Experiment 1

The end-point of the reaction is the first appearance of a blue colour.

Note: Insufficient swirling of the reaction mixture may lead to a blue colour appearing before the true end-point is reached.

1. Place the beaker containing **solution 1** on a white tile.
2. Using an appropriate measuring cylinder, transfer 20.0 cm^3 of **FA 6** to the beaker. Start the stopwatch during this addition.
3. Mix the contents in the beaker by **thoroughly** stirring with a glass rod.
4. Using a thermometer, note the initial temperature, T_i , of the reaction mixture.
5. Stop the stopwatch when the solution **first** turns blue.
6. Note the final temperature, T_f , of the reaction mixture.
7. Record the time taken, t , to the **nearest second**.
8. Wash the beaker and thermometer thoroughly with water and dry them.

(ii) Experiment 2

9. Prepare **solution 1** as described earlier.
10. Half-fill a 250 cm³ beaker with ice/water to make a cooling bath.
11. Place the 100 cm³ beaker, containing **solution 1**, into the cooling bath and carefully stir **solution 1** until its temperature is between 5 °C and 10 °C.
12. Remove the beaker from the cooling bath and repeat steps 2 – 8.

You have now performed the slowest experiment.

(iii) Experiment 3

13. Prepare **solution 1** as described earlier.
14. Half-fill a 250 cm³ beaker with hot water to make a heating bath.
15. Place the 100 cm³ beaker, containing **solution 1**, into the heating bath and carefully stir **solution 1** until its temperature is between 50 °C and 55 °C.
16. Remove the beaker from the heating bath and repeat steps 2 – 8.

You have now performed the fastest experiment.

(iv) Experiments 4 and 5

Select **two** other suitable reaction temperatures, between the temperatures of experiments 2 and 3, for use in two additional experiments. In each case, use the heating bath or cooling bath to bring **solution 1** to an appropriate temperature and perform these experiments.

Results

| expt | $T_i / ^\circ\text{C}$ | $T_f / ^\circ\text{C}$ | $T_{\text{avg}} / ^\circ\text{C}$ | T_K / K | t / s | $1/t / \text{s}^{-1}$ | $1/T_K / \text{K}^{-1}$ | $\ln(1/t)$ |
|------|------------------------|------------------------|-----------------------------------|------------------|----------------|-----------------------|-------------------------|------------|
| 1 | 31.0 | 31.0 | 31.0 | 304 | 34 | 0.0294 | 0.00328 | -3.53 |
| 2 | 10.0 | 14.0 | 12.0 | 285 | 105 | 0.00952 | 0.00351 | -4.65 |
| 3 | 51.0 | 45.0 | 48.0 | 321 | 14 | 0.0714 | 0.00312 | -2.64 |
| 4 | 22.0 | 24.0 | 23.0 | 296 | 54 | 0.0185 | 0.00338 | -3.99 |
| 5 | 44.0 | 40.0 | 42.0 | 315 | 19 | 0.0526 | 0.00317 | -2.94 |

Correct headings and units

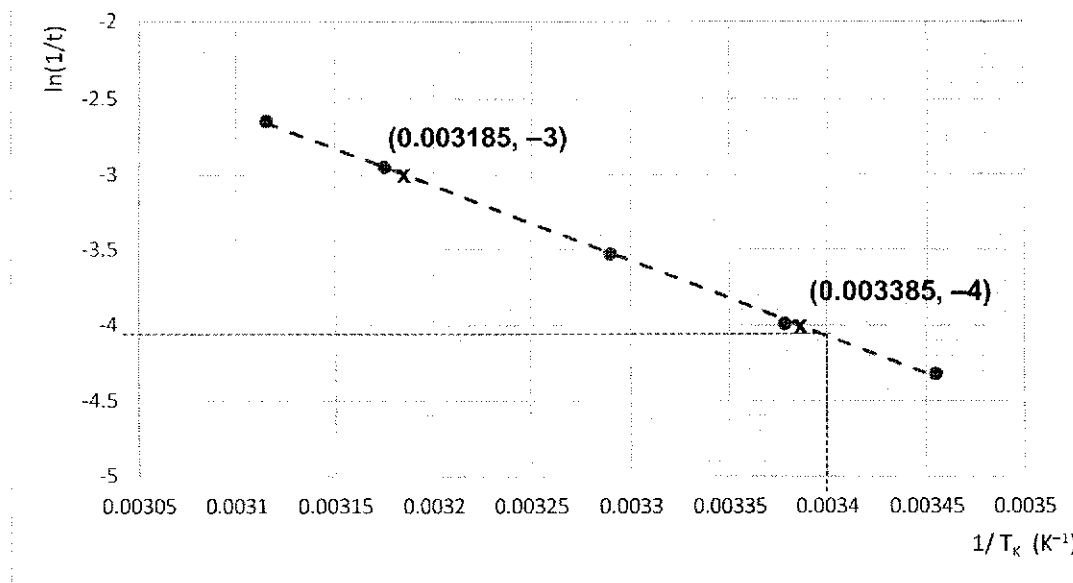
T to 0.1 °C, t to nearest second, calculated values 2-4 sf

t decreases as T_{avg} increases

Correct calculation of T_{avg} , T_K , $1/T_K$, $1/t$ and $\ln(1/t)$

[4]

- (b) (i) Plot a graph of $\ln(1/t)$ on the y-axis against $1/T_K$ on the x-axis. Draw the best-fit straight-line taking into account all of your plotted points.



Correct axis labels & graph more than $\frac{1}{2}$ of graph paper
 Correctly plotted points
 Best-fit straight-line

[3]

(ii) Calculate the gradient of the line, showing clearly how you did this.

$$\begin{aligned} \text{gradient} &= (-4 + 3) \div (0.003385 - 0.003185) \\ &= -5000 \text{ K} \end{aligned}$$

$$\text{gradient} = -5000 \text{ K} \quad [1]$$

(iii) Determine a value for the activation energy, E_a . Show your working.
 [$R = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$]

$$\begin{aligned} E_a &= -(-5000) \times 8.314 \\ &= 41.6 \text{ kJ mol}^{-1} \end{aligned}$$

$$\text{activation energy, } E_a = 41.6 \text{ kJ mol}^{-1} \quad [1]$$

(c) This version of the 'Iodine Clock' experiment is very reliable. With care, it is possible to perform a reaction that will change colour after a specified time interval.

Using **your graph**, calculate the temperature, in $^{\circ}\text{C}$, of the reaction mixture that would result in the reaction mixture turning blue after **58 seconds**. Show your working.

$$\ln(1/58) = -4.06 \approx -4.05$$

$$\begin{aligned} \text{From graph,} \\ 1/T_K &= 0.0034 \text{ K}^{-1} \\ T_K &= 294 \text{ K} \\ T &= 21.0 \text{ }^{\circ}\text{C} \end{aligned}$$

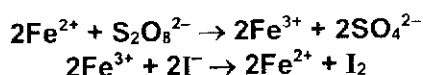
Note: Give proper working statements, correct sf and units in Q1 & Q2

$$\text{temperature required} = 21.0 \text{ }^{\circ}\text{C} \quad [3]$$

- (d) Another student performed **experiment 1**. She repeated the experiment but, before adding the **FA 6** solution, she added a few drops of **FA 3** (from Question 1) to **solution 1**. She found that this addition considerably reduced the time required for a dark blue colour to form.

With the aid of relevant equations, explain the mode of action **FA 3** in the reaction between peroxodisulfate and iodide ions.

The reaction between peroxodisulfate and iodide ions has high activation energy due to interelectronic repulsion between the two negatively charged ions.



FA 3 acts as a homogenous catalyst which provides an alternative pathway with lower activation. More particles have energy higher than activation energy, frequency of effective collisions increases and rate increases.

[2]

[Total: 14]

3 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 3.1** and **3.2**.

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) Inorganic analysis

You are provided with the solutions, **FA 7** and **FA 8**.

FA 7 is a solution containing one cation and one anion. The cation is singly charged and is **not** listed in the Qualitative Analysis notes.

FA 8 is prepared in the following way:

A metal oxide, M_2O , is added in excess to **FA 7**. A disproportionation reaction occurs, with a red-brown solid formed as one of the products. The red-brown solid, together with excess M_2O , is then removed via filtration. The filtrate collected is **FA 8**.

Table 3.1

| tests | | observations |
|-------|---|--|
| (i) | Add 1 cm depth of FA 7 in a test-tube, followed by 1 cm depth of aqueous sodium carbonate. | effervescence observed CO₂ gas, gives a white ppt with limewater |

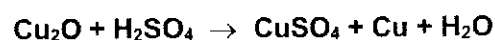
| | | |
|-------|---|---|
| (ii) | <p>Add 1 cm depth of FA 7 in a test-tube.</p> <p>Add aqueous barium chloride, followed by aqueous hydrochloric acid.</p> | <p>white ppt., insoluble in HCl</p> |
| (iii) | <p>Add 1 cm depth of FA 8 to a test-tube.</p> <p>Add aqueous ammonia slowly, with shaking, until no further change is seen.</p> <p>Then add FA 7 slowly, with shaking, until no further change is seen.</p> | <p>blue ppt., soluble in excess giving deep blue solution</p> <p>blue ppt. reformed, soluble in excess giving light blue solution</p> |
| (iv) | <p>Add 1 cm depth of FA 8 in a boiling tube, followed by 1 cm depth of aqueous sodium carbonate.</p> <p>Heat the mixture for about half a minute.</p> <p>You do not need to test for any gas.</p> | <p>blue (or green) ppt. formed</p> <p>ppt. turns black (or brown) on heating</p> |

[4]

(b) (i) Suggest the identity of the following ions:

Cation present in **FA 7** $\underline{\text{H}^+}$ (or H_3O^+)Anion present in **FA 7** $\underline{\text{SO}_4^{2-}}$ Cation present in **FA 8** $\underline{\text{Cu}^{2+}}$

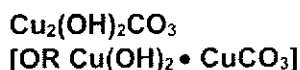
[2]

(ii) Hence, construct the full equation for the reaction between M_2O and **FA 7** to form **FA 8**.

[1]

(iii) When aqueous sodium carbonate was added to **FA 8** in (a)(iv), compound **R** is formed as one of the products. Compound **R** contains the cation of **FA 8** and two anions, **S** and **T**. The cation, **S** and **T** in compound **R** are present in the molar ratio of 2 : 2 : 1 respectively.

By considering the ions originally present in aqueous sodium carbonate and your answer in (b)(i), suggest the formula of the product **R**.

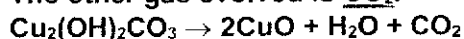


[1]

- (iv) Two gases were evolved during heating in (a)(iv) but it is difficult for you to detect them due to the short duration of the heating process. One of the gases evolved was water vapour.

Suggest the identity of the other gas evolved. Hence, construct an equation for the reaction that took place during heating in (a)(iv).

The other gas evolved is CO_2 .



[1]

(c) **Organic analysis**

In this question, you will deduce the structure of an organic compound, **FA 9**.

FA 9 has the molecular formula $\text{C}_4\text{H}_8\text{O}_2$ 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 3.2

| | tests | observations |
|------|---|---|
| (i) | Place about 2 cm depth of aqueous sulfuric acid in a test-tube. To this test-tube, add 15 drops of FA 9 , followed by 1 drop of aqueous potassium manganate(VII). Warm the mixture in the hot water bath for two minutes. | purple KMnO_4 decolourised |
| (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. You may use a clean glass rod to stir the mixture and help dissolve the precipitate. | brown (or grey) ppt. formed (ppt. dissolves) |

| | | |
|-------|---|--|
| | <p>To this mixture, add 10 drops of FA 9.</p> <p>Place 50 cm³ of hot water in a beaker and note its temperature. If the temperature is higher than 60 °C, add a small amount of water at room temperature, stir and note the temperature.</p> <p>Keep adding small amounts of water until the temperature of the water is 60 °C or lower.</p> <p>Place the test-tube containing the mixture in the water bath for one minute.</p> | <p>no observable change [OR no silver mirror]</p> |
| (iii) | <p>Place about 1 cm depth of FA 9 in a test-tube.</p> <p>To this test-tube, add 2,4-dinitrophenylhydrazine dropwise.</p> | <p>orange ppt. formed</p> |
| (iv) | <p>Place about 15 drops of FA 9 and add 10 drops of aqueous sodium hydroxide in a test-tube.</p> <p>Now add iodine solution dropwise, until a permanent orange/red colour is obtained.</p> <p>Warm the mixture in the hot water bath for two minutes.</p> | <p>pale yellow ppt. formed</p> |
| (v) | <p>Place 1 cm depth of FA 9 in a test-tube.</p> <p>To this test-tube, cautiously add a small piece of sodium metal.</p> | <p>effervescence observed H₂ gas produced extinguishes a lighted splint with a "pop" sound.</p> |

[2]

- (d) (i) Observations from (c)(iii), together with those of **either (c)(i) or (c)(ii)**, can be used to identify one of the functional groups present in **FA 9**.

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

Functional group: **ketone**

Explanation:

Since FA 9 undergoes condensation with 2,4-DNPH in (c)(iii), FA 9 contains either a ketone or aldehyde (or carbonyl compound).

However, since FA 9 gives a negative test with Tollens' reagent in (c)(ii), it does not contain an aldehyde.

[1]

- (ii) Using observations from **either (c)(iv) or (v)**, identify the other functional group present in FA 9.

Quote evidence from the relevant test to support your conclusion.

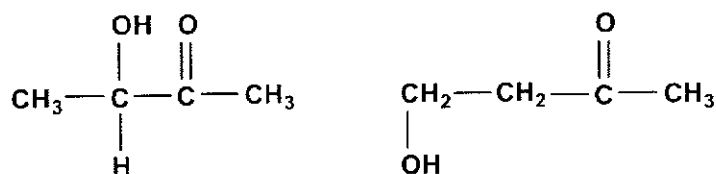
Functional group: alcohol

Evidence:

Since FA 9 undergoes acid-metal reaction with Na in (c)(v) to give H₂, it contains an alcohol functional group.

[1]

- (iii) Suggest two possible structures of FA 9 that are consistent with all the observations in Table 3.2.



[2]

[Total: 15]

4 Planning

FA 10 is a solution of aqueous potassium hydroxide, KOH, with a rough concentration of 0.2 mol dm^{-3} . FA 11 is a mixture of $0.100 \text{ mol dm}^{-3}$ aqueous sulfuric acid, H₂SO₄, and $0.100 \text{ mol dm}^{-3}$ sodium sulfate, Na₂SO₄.

To determine the accurate concentration of potassium hydroxide in FA 10, a series of experiments can be conducted in which the temperature change in each experiment is recorded. In each experiment, a different volume of FA 11 is used but the volume of FA 10 is kept constant. The total volume of the reaction mixture is also kept constant by adding an appropriate volume of water.

A graph of *temperature change* against the *volume of acid added* is then plotted. In this graph, two best-fit lines are drawn and extrapolated until they cross one another. The point of intersection is the equivalence point of the chemical reaction.

With the equivalence point determined from the graph, the accurate concentration of aqueous potassium hydroxide can then be determined.

- (a) Using the information given above, write an experimental plan to determine the equivalence point for the reaction between FA 10 and FA 11.

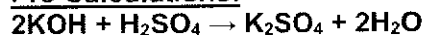
You may assume that you are provided with:

- FA 10, which is a solution of KOH with a rough concentration 0.2 mol dm^{-3} ,
- FA 11, which is a mixture of $0.100 \text{ mol dm}^{-3}$ H₂SO₄ and $0.100 \text{ mol dm}^{-3}$ Na₂SO₄,
- the equipment normally found in a school or college laboratory.

In your plan, you should include brief details of:

- calculation of the approximate volume of FA 11 required to completely neutralise a fixed volume of 20.0 cm³ of FA 10,
- a table containing the volumes of each reagent to be added for six experiments,
- the apparatus you would use,
- the procedure you would follow,
- the measurements you would make to allow a graph of *temperature change* against the *volume of acid added* to be drawn.

Pre-Calculations:



$$\begin{aligned} \text{Amount of KOH} &= 20.0/1000 \times 0.2 \\ &= 0.00400 \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Amount of H}_2\text{SO}_4 &= 1/2 \times 0.00400 \text{ mol} \\ &= 0.00200 \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Volume of H}_2\text{SO}_4 &= (0.00200 / 0.100) \times 1000 \\ &= 20.0 \text{ cm}^3 \text{ (approximate)} \end{aligned}$$

Table:

| Experiment | Volume of FA 10 / cm ³ | Volume of FA 11 (acid) / cm ³ | Volume of H ₂ O / cm ³ |
|------------|-----------------------------------|--|--|
| 1 | 20.0 | 5.0 | 55.0 |
| 2 | 20.0 | 10.0 | 50.0 |
| 3 | 20.0 | 15.0 | 45.0 |
| 4 | 20.0 | 20.0 | 40.0 |
| 5 | 20.0 | 30.0 | 30.0 |
| 6 | 20.0 | 40.0 | 20.0 |

Volume of FA 10 (KOH) in each mixture is fixed (20.0 cm³) and total volume of six reaction mixtures is fixed and less than 200 cm³ (to ensure that total volume of solution does not exceed capacity of Styrofoam cup)
At least 3 volumes less than equivalence volume (20.0 cm³)

Procedure:

1. Using a measuring cylinder, transfer 20.0 cm³ of FA 10 into a Styrofoam cup supported by a beaker.
2. Using another measuring cylinder, measure the 55.0 cm³ of H₂O and add it into the Styrofoam cup.
3. Using a thermometer, record the initial temperature of the mixture in the Styrofoam cup.
4. Using another measuring cylinder, measure the 5.0 cm³ of FA 11.
5. Using a thermometer, record the initial temperature of FA 11.
6. Transfer the FA 11 into the Styrofoam cup and stir using a glass rod.
7. Using the thermometer, record the highest temperature reached.
8. Repeat steps 1 to 7 by using the volumes indicated in the table above to obtain five more sets of data.

[5]

- (b) (i) Sketch on Figure 4.1 the graph you would expect to get from your results.

Indicate on the graph the equivalence point, V (in cm³) and the maximum temperature rise, A.

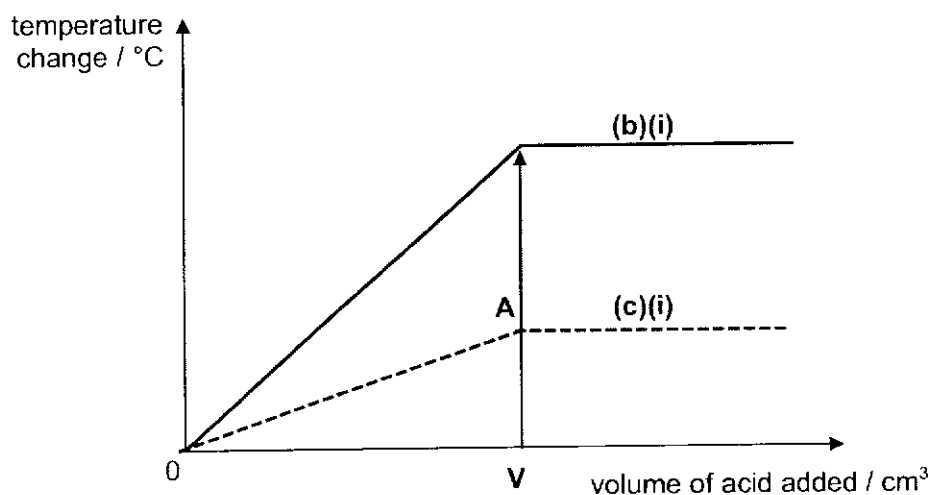
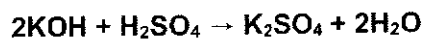


Figure 4.1

Graph with correct shape and starts from 0
Correct labelling of A and V on the graph

[1]

- (ii) Show how the data obtained from the graph in (b)(i) can be used to determine
- the accurate concentration of potassium hydroxide, and
 - enthalpy change of neutralisation, ΔH_n in J mol^{-1} , assuming x J is required to raise the temperature of 1 cm^3 of solution by 1°C and the total volume of the mixture is $V_t \text{ cm}^3$



$$\text{Volume of KOH used} = 20.0 \text{ cm}^3$$

$$\text{Volume of H}_2\text{SO}_4 \text{ to completely neutralise KOH} = V \text{ cm}^3$$

$$\begin{aligned} \text{Amount of H}_2\text{SO}_4 \text{ used} &= (V/1000) \times 0.100 \\ &= 1.00 \times 10^{-4} V \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Amount of KOH reacted} &= 1.00 \times 10^{-4} V \times 2 \\ &= 2.00 \times 10^{-4} V \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{Concentration of KOH} &= 2.00 \times 10^{-4} V / 20.0 \times 10^{-3} \\ &= 0.0100 V \text{ mol dm}^{-3} \end{aligned}$$

$$\begin{aligned} \text{Heat evolved} &= V_{\text{total}} c \Delta T \\ &= V_t \times A \text{ J} \end{aligned}$$

$$\begin{aligned} \text{Amount of H}_2\text{O formed} &= \text{Amount of KOH reacted} \\ &= 2.00 \times 10^{-4} V \text{ mol} \end{aligned}$$

$$\Delta H_n = -[V_t \times A] \div 2.00 \times 10^{-4} V \text{ J mol}^{-1}$$

[2]

(c) In another experiment, you are given **FA 12**, which is a mixture of $0.100 \text{ mol dm}^{-3}$ aqueous ethanedioic acid, $\text{HO}_2\text{CCO}_2\text{H}$ and $0.1000 \text{ mol dm}^{-3}$ sodium ethanedioate, $\text{NaO}_2\text{CCO}_2\text{Na}$.

(i) Sketch on **Figure 4.1** another graph to show the results you would expect to obtain when **FA 12** is used instead of **FA 11**. Label the graph with **(c)(i)**.

For (c)(i), line rises less and intersects second line at a lower temperature rise at the same equivalence-point volume.

[1]

(ii) Explain the difference in the two graphs in **Figure 4.1**.

$\text{HO}_2\text{CCO}_2\text{H}$ is a weak acid that partially dissociates to give H^+ . Some of the heat evolved during neutralisation will be used to completely dissociate the undissociated $\text{HO}_2\text{CCO}_2\text{H}$ molecules. Thus, the maximum temperature rise will be lower.

Note: Both $\text{HO}_2\text{CCO}_2\text{H}$ and H_2SO_4 are dibasic acids. Same amount of acid will be required for complete neutralisation. Thus, the equivalence point will remain unchanged.

[2]

[Total: 11]

