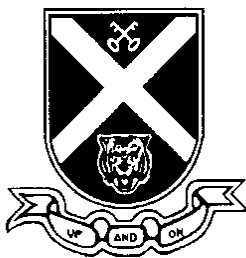


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|-------|--|--------|--|
| Name: |  | Class: |  |
|-------|--|--------|--|

## ST ANDREW'S JUNIOR COLLEGE



## JC 2 PRELIMINARY EXAMINATION

**CHEMISTRY**

**9729/04**

**Paper 4 Practical**

**16 Aug 2021**

**2 hours 30 minutes**

**Additional Materials: Qualitative Analysis Notes**

### READ THESE INSTRUCTIONS FIRST.

Write your name and class on all the work you hand in.

Give details of the practical shift and laboratory in the boxes provided above.

Write in dark blue or black pen.

You may use a soft pencil for any diagrams or graphs.

Do not use staples, paper clips, highlighters, 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.**

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

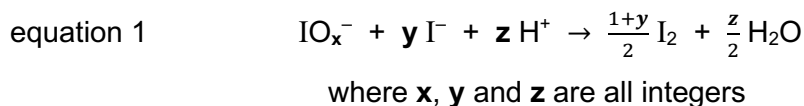
|                   |
|-------------------|
| <b>Shift</b>      |
|                   |
| <b>Laboratory</b> |
|                   |

| For Examiner's Use |    |
|--------------------|----|
| 1                  | 13 |
| 2                  | 18 |
| 3                  | 10 |
| 4                  | 14 |
| Total              | 55 |

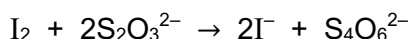
1. **Determination of the value of  $x$  in the oxyanion of iodine,  $\text{IO}_x^-$**

Iodine is able to form more than one oxyanion,  $\text{IO}_x^-$ , polyatomic ions that contain oxygen, each containing a different number of oxygen atoms.

In this experiment, you will determine the value of  $x$  in the oxyanion of iodine,  $\text{IO}_x^-$ . You will first react  $\text{IO}_x^-$  ions with an excess of iodide ions,  $\text{I}^-$ , to form iodine,  $\text{I}_2$  as shown in equation 1.



The amount of iodine produced will then be determined by titration with thiosulfate ions,  $\text{S}_2\text{O}_3^{2-}$ .



**FA 1** is a solution containing  $0.0150 \text{ mol dm}^{-3}$   $\text{IO}_x^-$  ions.

**FA 2** is dilute sulfuric acid,  $\text{H}_2\text{SO}_4$ .

**FA 3** is  $1.00 \text{ mol dm}^{-3}$  potassium iodide,  $\text{KI}$ .

**FA 4** is  $0.100 \text{ mol dm}^{-3}$  sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ .

starch indicator

**(a) Procedure**

1. Fill the burette with **FA 4**.
2. Pipette  $25.0 \text{ cm}^3$  of **FA 1** into a conical flask.
3. Use a measuring cylinder to add  $25 \text{ cm}^3$  of **FA 2** to the conical flask.
4. Use another measuring cylinder to add  $10 \text{ cm}^3$  of **FA 3** to the conical flask.  
The solution will turn brown as iodine is produced.
5. Add **FA 4** from the burette until the solution in the conical flask turns yellow.
6. Add 5 drops of starch indicator to the conical flask. The solution will turn blue-black.
7. Continue to add more **FA 4** from the burette until the blue-black colour just disappears. This is the end-point of the titration.
8. Record your titration results, to an appropriate level of precision, in the space provided on page 3.
9. Repeat points 2 to 7 until consistent results are obtained.

**Results****[3]**

|                                          |       |       |  |
|------------------------------------------|-------|-------|--|
| Initial burette reading /cm <sup>3</sup> | 0.00  | 0.00  |  |
| Final burette reading /cm <sup>3</sup>   | 25.60 | 25.70 |  |
| Volume of <b>FA 4</b> /cm <sup>3</sup>   | 25.60 | 25.70 |  |

- Correct headers, units and final burette reading greater than initial burette reading
- Correct precision to 2 d.p
- 2 consistent values within  $\pm 0.10 \text{ cm}^3$

- (b) From your titrations, obtain a suitable volume of **FA 4**, to be used in your calculations. Show clearly how you obtained this value. **[2]**

$$\text{Volume of FA 4} = \frac{25.60 + 25.70}{2} = 25.65 \text{ cm}^3$$

- Correct calculation of average titre volume to 2 d.p and correct calculation of volume of **FA 4** in the titration table
- Accuracy

volume of **FA 4** = .....

- (c) (i) Calculate the number of moles of iodine formed when **FA 1** reacts with **FA 3**. **[1]**

$$\begin{aligned} \text{Number of moles of I}_2 &= \frac{1}{2} \times (25.65 / 1000 \times 0.1) \\ &= 1.28 \times 10^{-3} \end{aligned}$$

number of moles of  $\text{I}_2$  = .....

- (ii) Calculate the number of moles of  $\text{IO}_x^-$  ions in  $25.0 \text{ cm}^3$  of **FA 1**. **[1]**

$$\text{Number of moles of IO}_x^- \text{ ions} = 25 / 1000 \times 0.015 = 3.75 \times 10^{-4}$$

number of moles of  $\text{IO}_x^-$  ions = .....

**[Turn Over]**

- (iii) Using equation 1 and your answers in **1(c)(i)** and **1(c)(ii)**, calculate **[1]**  
the value of **y**. Show your working.  
(Note that **y** is an odd integer such as 1, 3, 5, 7 etc.)

$$\frac{1.28 \times 10^{-3}}{3.75 \times 10^{-4}} = \frac{1+y}{2}$$

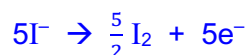
$$y = 5.83 \approx 5$$

**y** = .....

- (iv) Use your value of **y** in **1(c)(iii)** and considering the number of **[2]**  
electrons transferred, determine the oxidation number of I in  $\text{IO}_x^-$  ion.  
Hence, determine the value of **x** in  $\text{IO}_x^-$  ion.



2 moles of  $\text{I}^-$  gives out 2 moles of electrons



5 moles of  $\text{I}^-$  gives out 5 moles of electrons

1 mole of  $\text{IO}_x^-$  gains 5 moles of electrons to form  $\text{I}_2$

Hence, oxidation number of I in  $\text{IO}_x^-$  is +5

$$+5 + x(-2) = -1$$

$$x = 3$$

oxidation number of I in  $\text{IO}_x^-$  ion = .....

**x** = .....

- (v) A student suggested that a more accurate value of **x** could be **[1]**  
obtained if a  $10.0 \text{ cm}^3$  pipette was used to measure **FA 3** rather than  
the measuring cylinder.  
State whether you agree with the student. Explain your answer.  
I do not agree as KI is in excess so precision of the apparatus does  
not matter.

- (vi) Explain how the titre volume of  $\text{Na}_2\text{S}_2\text{O}_3$  will change when the value of  $x$  in  $\text{IO}_x^-$  is greater. [1]

When  $x$  is greater, higher amount of iodine will be produced, which require a higher titre volume of  $\text{S}_2\text{O}_3^{2-}$ .

- (d) Chlorine is also able to form more than one oxyanion,  $\text{ClO}_x^-$ . [1]

Similar to  $\text{IO}_x^-$ , oxyanions of chlorine are also oxidising agents. The table below shows the standard electrode potential of different chlorine oxyanions.

| Electrode Reaction                                                                                 | $E^\circ / \text{V}$ |
|----------------------------------------------------------------------------------------------------|----------------------|
| $\text{ClO}^- + \text{H}_2\text{O} + 2\text{e}^- \rightleftharpoons \text{Cl}^- + 2\text{OH}^-$    | +0.89                |
| $\text{ClO}_2^- + 2\text{H}_2\text{O} + 4\text{e}^- \rightleftharpoons \text{Cl}^- + 4\text{OH}^-$ | +0.78                |
| $\text{ClO}_3^- + 3\text{H}_2\text{O} + 6\text{e}^- \rightleftharpoons \text{Cl}^- + 6\text{OH}^-$ | +0.63                |
| $\text{ClO}_4^- + 4\text{H}_2\text{O} + 8\text{e}^- \rightleftharpoons \text{Cl}^- + 8\text{OH}^-$ | +0.56                |
| $\text{Cl}_2 + 2\text{e}^- \rightleftharpoons 2\text{Cl}^-$                                        | +1.36                |

However, unlike  $\text{IO}_x^-$ , the value of  $x$  in the oxyanion of chlorine,  $\text{ClO}_x^-$  cannot be determined by reacting  $\text{ClO}_x^-$  with its corresponding halide,  $\text{Cl}^-$ .

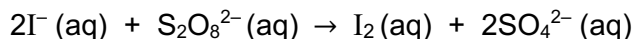
Use the data given in the above table, explain why it is not possible to determine the value of  $x$  in the oxyanion of chlorine,  $\text{ClO}_x^-$ , with the reaction with  $\text{Cl}^-$ , under standard conditions.

$E^\circ_{\text{cell}}$  is negative for all reactions between  $\text{ClO}_x^-$  and  $\text{Cl}^-$ , hence reaction is not spontaneous / feasible.

[Total: 13]

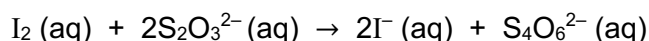
## 2. Investigation of the effect of $[\text{S}_2\text{O}_8^{2-}]$ on the rate of reaction between $\text{I}^-$ and $\text{S}_2\text{O}_8^{2-}$

Sulfur forms the peroxodisulfate anion,  $\text{S}_2\text{O}_8^{2-}$ . This ion can oxidise iodide ions,  $\text{I}^-$ , to iodine,  $\text{I}_2$ , as shown in the equation.



You will carry out a series of experiments to investigate how the rate of this reaction is affected by changing the concentration of the solutions.

The rate can be measured by adding thiosulfate ions,  $\text{S}_2\text{O}_3^{2-}$ , and starch indicator. As the reaction between  $\text{S}_2\text{O}_8^{2-}$  and  $\text{I}^-$  occurs, iodine is produced. The  $\text{I}_2$  produced reacts immediately with the thiosulfate.



When all the thiosulfate has reacted, the iodine will remain in the mixture and cause the starch indicator to turn blue-black. The rate of reaction may be determined by measuring the time taken for the reaction mixture to turn blue-black.

**FA 5** is  $0.0200 \text{ mol dm}^{-3}$  potassium peroxodisulfate,  $\text{K}_2\text{S}_2\text{O}_8$ .

**FA 6** is  $1.00 \text{ mol dm}^{-3}$  potassium iodide,  $\text{KI}$ .

**FA 7** is  $0.00500 \text{ mol dm}^{-3}$  sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ .

starch indicator

### (a) (i) Procedure

#### Experiment 1

1. Use the marker to label one of the  $100 \text{ cm}^3$  beakers '**A**' and the other  $100 \text{ cm}^3$  beaker '**B**'.
2. Use the marker to label one of the measuring cylinders '**A**' and the other measuring cylinder '**B**'.
3. Use the measuring cylinder **A** to transfer  $20.0 \text{ cm}^3$  of **FA 5** into beaker **A**.
4. Use the measuring cylinder **B** to add  $20.0 \text{ cm}^3$  of **FA 6** into beaker **B**.
5. Use the measuring cylinder **B** to add  $10.0 \text{ cm}^3$  of **FA 7** to beaker **B**.
6. Add 10 drops of starch indicator to beaker **B**.
7. Add the contents of beaker **A** to beaker **B**. Start the stopwatch during this addition.

8. Stir the mixture **once** and place the beaker on a white tile.
9. Stop the stopwatch when the solution **first** turns blue-black.
10. Record this reaction time to the nearest second.
11. Wash out both beakers and shake to remove excess water.

### Experiment 2

1. Use the measuring cylinder **A** to transfer 10.0 cm<sup>3</sup> of **FA 5** into beaker **A**.
2. Use the measuring cylinder labelled **A** to transfer 10.0 cm<sup>3</sup> of distilled water into beaker **A**.
3. Use the measuring cylinder **B** to add 20.0 cm<sup>3</sup> of **FA 6** into beaker **B**.
4. Use the measuring cylinder **B** to add 10.0 cm<sup>3</sup> of **FA 7** to beaker **B**.
5. Add 10 drops of starch indicator to beaker **B**.
6. Add the contents of beaker **A** to beaker **B**. Start the stopwatch during this addition.
7. Stir the mixture once and place the beaker on a white tile.
8. Stop the stopwatch when the solution **first** turns blue-black.
9. Record this reaction time to the nearest second.
10. Wash out both beakers and shake to remove excess water.

### Experiments 3 to 5

Choose suitable volumes that will enable you to investigate further the effect of changing the concentration of potassium peroxodisulfate, **FA 5**, on the rate of the reaction.

Note that the total volume of **FA 5** and distilled water must always be constant. Do not use a volume of **FA 5** that is less than 6.0 cm<sup>3</sup>.

The relative rate of the reaction can be calculated as shown.

$$\text{Relative rate} = \frac{900}{\text{reaction time}}$$

In the space provided, record in a single table for each of your five experiments:

- volume of **FA 5**
- volume of distilled water
- the reaction time
- the relative rate of the reaction

**Results**

| Expt | Volume of<br>FA 5 / cm <sup>3</sup> | Volume of<br>distilled water<br>/ cm <sup>3</sup> | Time /<br>s | Relative<br>Rate / s <sup>-1</sup> |
|------|-------------------------------------|---------------------------------------------------|-------------|------------------------------------|
| 1    | 20.0                                | 0.0                                               | 15          | 60.0                               |
| 2    | 10.0                                | 10.0                                              | 30          | 30.0                               |
| 3    | 15.0                                | 5.0                                               | 20          | 45.0                               |
| 4    | 12.0                                | 8.0                                               | 26          | 34.6                               |
| 5    | 8.0                                 | 12.0                                              | 36          | 25.0                               |

**[5]**

- Correct headers and units
- 3 additional volumes chosen  
Intervals not less than 2.0 cm<sup>3</sup> and  
All volumes of **FA 5** between 6.0 – 20.0 cm<sup>3</sup>
- Correct volume of deionised water used and rate calculated
- Time taken increases with decreasing volume of **FA 5**
- Correct precision recorded:
  - Volume of **FA 5** to 1 d.p
  - Volume of deionised water to 1 d.p
  - Time to whole number
  - rate to 3 s.f.

- (ii) Using data from **Experiments 1** and **2**, show by calculation that the volume of potassium peroxodisulfate, **FA 5**, used was directly proportional to the concentration of peroxodisulfate. You can ignore the volume of starch used. **[2]**

Expt 1:

Since  $C_1V_1 = C_2V_2$  $C_1 = 0.02 \text{ mol dm}^{-3}$  and  $V_1 = 20 \text{ cm}^3$  and  $V_2 = 50 \text{ cm}^3$ 

$$\Rightarrow [\text{S}_2\text{O}_8^{2-}] = 2/5 \times 0.02 = 8.00 \times 10^{-3} \text{ mol dm}^{-3}$$

Expt 2:

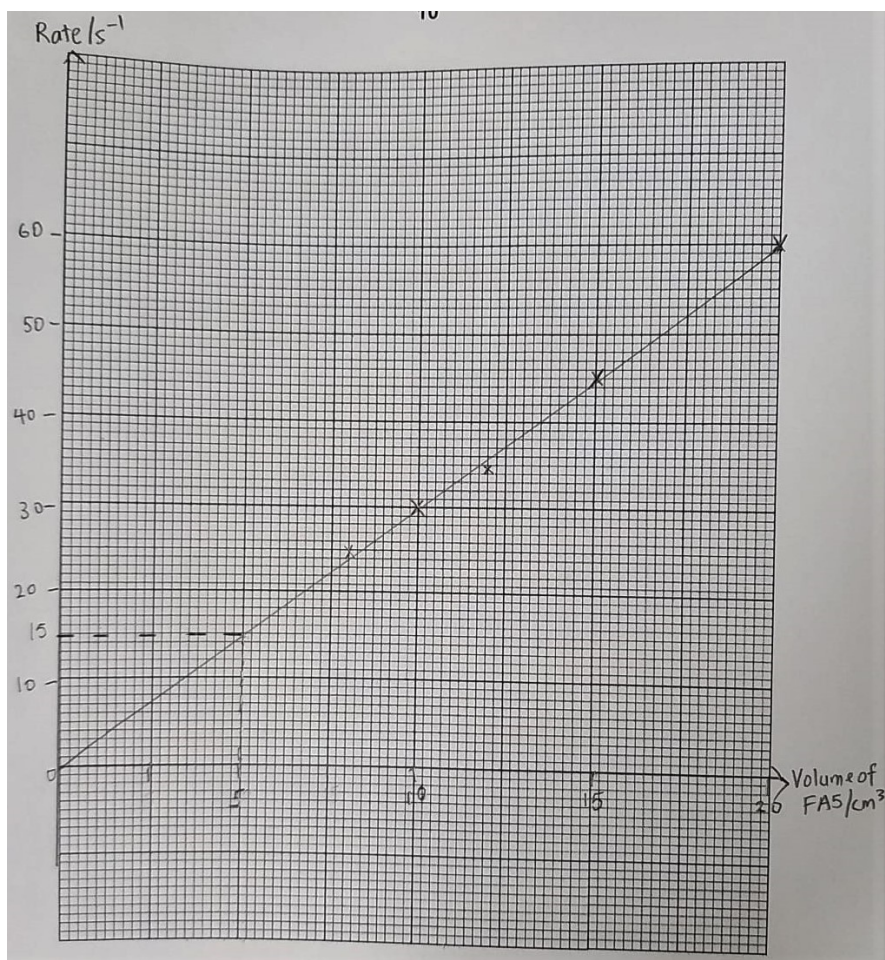
Since  $C_1V_1 = C_2V_2$  $C_1 = 0.02 \text{ mol dm}^{-3}$  and  $V_1 = 10 \text{ cm}^3$  and  $V_2 = 50 \text{ cm}^3$ 

$$\Rightarrow [\text{S}_2\text{O}_8^{2-}] = 1/5 \times 0.02 = 4.00 \times 10^{-3} \text{ mol dm}^{-3}$$

**[Turn Over]**

When volume is doubled from 10 to 20 cm<sup>3</sup>, [S<sub>2</sub>O<sub>8</sub><sup>2-</sup> is doubled,  
hence volume of S<sub>2</sub>O<sub>8</sub><sup>2-</sup> is directly proportional to [S<sub>2</sub>O<sub>8</sub><sup>2-</sup>].

- (b) (i) Use the grid below to plot a graph of rate against volume of FA 5. [3]  
Include the origin in your plot.



- Linear scales cover more than half the space in both directions including (0, 0).
- Axes correctly orientated and clearly labelled.
- Points plotted correctly.
- Points must be within half a small square of the correct position.
- Line of best fit drawn which ignores anomalous results identified by the candidate.
- The line may be straight or smooth curve AND use a minimum of 3 points.
- Reject if a point has been shown at the origin and the line of best fit does not pass within 5 small squares of (0,0).

- (b) (ii) Explain, by referring to your graph or your table of results, how the rate of reaction is affected by an increase in the concentration of potassium peroxodisulfate, **FA 5**. [1]

Straight line: Rate is proportional to concentration (of iodide ions) / proportional as line has a positive gradient.

Curve: As concentration / volume (of iodide ions) increases, rate increases more / not directly proportional as line is a curve / not a straight line.

OR

Table: Compares ratio of concentrations / volumes of FA 1 with ratio of rates. Directly proportional as rate doubled when volume doubled.

- (iii) Use your graph to calculate the reaction time you would expect to measure if you carried out an experiment using 5.00 cm<sup>3</sup> of **FA 5**. [2]  
Show on the graph how you obtained your answer.

From graph, when volume = 5 cm<sup>3</sup>, rate = 15.0 s<sup>-1</sup>

$$\begin{aligned}\text{Reaction time} &= \frac{900}{\text{rate}} \\ &= \frac{900}{15} \\ &= 60.0 \text{ s}\end{aligned}$$

reaction time = .....

- (iv) Assume that the error in the time measured for each reaction was ±0.5 s in total. Calculate the percentage error in the reaction time you measured in **Experiment 1**. Show your working. [2]

$$\% \text{ error} = \frac{\pm 0.5}{15} \times 100 \% = \pm 3.33 \%$$

percentage error = .....

- (v) A student suggested that this error could be reduced if 0.00200 mol dm<sup>-3</sup> sodium thiosulfate was used in place of **FA 7**. Do you agree with this student? Explain your answer. [1]

The student is wrong as the reaction time will be shorter and hence the percentage error would be greater.

- (vi) A student carries out the same investigation as in **2(a)(i)** but the solutions are mixed in a different order. The student places **FA 5** and an appropriate volume of distilled water in beaker **A**. He then added **FA 7** and starch into beaker **A**. He added **FA 6** last and started the stopwatch immediately. [2]

Tick the box for the statement you consider correct. Explain your answer.

|                                                             |                          |
|-------------------------------------------------------------|--------------------------|
| The student's method is better than that in <b>(a)</b> .    | <input type="checkbox"/> |
| The two methods are equally good.                           | <input type="checkbox"/> |
| The student's method is not as good as that in <b>(a)</b> . | <input type="checkbox"/> |

You may use the data in the table below to explain your answer.

| Electrode Reaction                                                                      | E° / V |
|-----------------------------------------------------------------------------------------|--------|
| $\text{S}_2\text{O}_8^{2-} + 2\text{e}^- \rightleftharpoons 2\text{SO}_4^{2-}$          | +2.01  |
| $\text{S}_4\text{O}_6^{2-} + 2\text{e}^- \rightleftharpoons 2\text{S}_2\text{O}_3^{2-}$ | +0.09  |
| $\text{I}_2 + 2\text{e}^- \rightleftharpoons 2\text{I}^-$                               | +0.54  |

Reason: .....

.....  
.....

Not as good.

S<sub>2</sub>O<sub>8</sub><sup>2-</sup> will react with S<sub>2</sub>O<sub>3</sub><sup>2-</sup> and less S<sub>2</sub>O<sub>3</sub><sup>2-</sup> will be left in the reaction mixture to react with iodine. Hence, time will decrease for each run.

OR

S<sub>2</sub>O<sub>8</sub><sup>2-</sup> will react with S<sub>2</sub>O<sub>3</sub><sup>2-</sup> and a lower concentration of S<sub>2</sub>O<sub>8</sub><sup>2-</sup> results in a slower rate of producing iodine. Hence, time will increase for each run.

[Total: 18]

[Turn Over]

### 3. Planning

A student suggested that the temperature at which the experiment was carried out would also affect the rate of the reaction.

- (a) Plan an investigation, based on the experiment described in 2(a)(i), to [4]  
determine the effect of temperature on the rate of reaction.

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

Give a step-by step description of how you would carry out the experiment by considering

- what you would keep constant in all the experiments,
  - a suitable number of experiments you would do, and a reasonable temperature range,
  - the apparatus that you would use in addition to that specified in 2(a)(i)
  - the procedure that you would follow and the measurements that you would take
  - how you would determine the rate for each experiment.
1. After step 2 and step 4 in 2(a)(i), place beaker A and beaker B in a temperature controlled water bath (A) at 10°C.
  2. Use a thermometer (A) to measure and ensure the temperature in beaker A and beaker B is the same as the water bath before mixing. (P)
  3. When both solutions have reached 10.0°C, add the contents of beaker A to beaker B and start timing immediately. (P)
  4. Repeat the experiment with the same volume of FA 5, FA 6, FA 7 and starch indicator (Q) used but at temperature 20.0°C, 30.0°C, 40.0°C, 50.0°C. (P, R)
  5. Calculate rate  $\propto \frac{1}{\text{time}}$  at each temperature. (P)

|               |                                                                                                            |
|---------------|------------------------------------------------------------------------------------------------------------|
| Procedure (P) | 1. Measure and ensure the temperature in beaker A and beaker B is the same as the water bath before mixing |
|---------------|------------------------------------------------------------------------------------------------------------|

|                 |                                                                                                                                                                                                              |
|-----------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|                 | 2. Add the contents of beaker <b>A</b> to beaker <b>B</b> and start timing immediately<br>3. Repeat the experiment at at temperature 20.0°C, 30.0°C, 40.0°C, 50.0°C<br>4. Calculate rate at each temperature |
| Apparatus (A)   | <ul style="list-style-type: none"> <li>• Temperature controlled water bath</li> <li>• Thermometer</li> </ul>                                                                                                 |
| Reliability (R) | <ul style="list-style-type: none"> <li>• Temperatures should be less than 100 °C and cover at least a 10°C range of temperatures</li> </ul>                                                                  |
| Quantity (Q)    | <ul style="list-style-type: none"> <li>• Same volume of <b>FA 5</b>, <b>FA 6</b>, <b>FA 7</b> and starch indicator</li> </ul> Credit if made reference to Q2 and repeated steps 1 – 6.                       |

- (b) 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 at reaction temperature  $T$  in Kelvin,  $A$  is the frequency factor and  $R$  is the ideal gas constant ( $8.314 \text{ J K}^{-1}\text{mol}^{-1}$ ).  $A$  can be regarded as a constant for this experiment.

Taking the natural logarithm of the "Arrhenius equation", gives the following equation.

$$\ln k = \ln A - \frac{E_a}{R} \left( \frac{1}{T_K} \right)$$

- (i) Given that rate =  $k$  [reactants], state the relationship between  $k$  and the time taken for the reaction mixture to turn blue-black. **[1]**

Since [reactants] are constant, rate  $\propto k$

Since rate  $\propto 1/t$ ,

$k \propto 1/t$

- (ii) Sketch a graph that you would use to determine the activation energy,  $E_A$ , for the reaction on the axes in **Figure 3.1**. Describe how you would use your graph to determine the value of  $E_A$ . [3]

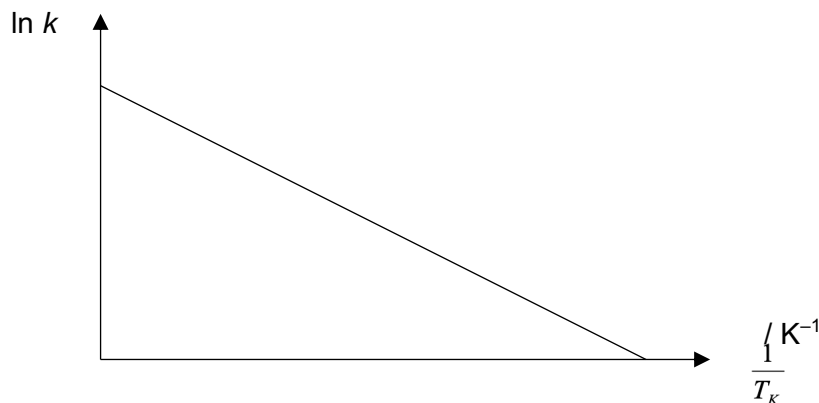


Figure 3.1

Calculate  $E_a$  by taking two sets of co-ordinates on the graph ( $x_1, y_1$ ) and ( $x_2, y_2$ )

Find the gradient =  $\frac{y_1 - y_2}{x_1 - x_2}$

Equate  $\frac{y_1 - y_2}{x_1 - x_2} = -\frac{E_a}{R}$  and determine  $E_a$  by multiplying  $-R$  with gradient.

- (iii) Briefly describe how you would use results obtained from **3(a)** to determine all necessary values in order to plot the graph in **3(b)(ii)**. You do **not** need to calculate any of the calculations. [2]

Since time  $\propto \frac{1}{\text{rate}}$  and rate  $\propto$  rate constant,  $k$ , calculate  $\ln k$  by taking

$\ln \left( \frac{1}{t} \right)$

Convert  $T/^{\circ}\text{C}$  to  $\text{T/K}$  and calculate  $\frac{1}{T_k}$

| Expt | Time / s | $\ln k \propto \ln \left( \frac{1}{t} \right)$ | $T/^{\circ}\text{C}$ | $\text{T/K}$ | $\frac{1}{T_k} \text{ K}^{-1}$ |
|------|----------|------------------------------------------------|----------------------|--------------|--------------------------------|
|      |          |                                                |                      |              |                                |

[Total: 10]

[Turn Over

#### 4. Investigation of some inorganic and organic reactions

(a) **FA 8** is a mixture that contains two cations and two anions.

Distilled water was added to **FA 8**, where the mixture was stirred and then filtered. You are provided with the dried residue, **FA 9**, and the filtrate, **FA 10**, from this filtration process.

Carry out the tests described in **Table 4.1** and carefully record your observations in the table.

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

**Table 4.1**

|    | tests                                                                                                                                                                                                                        | observations                                                                                                                                                                                                                                                   |
|----|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1. | (a) Add one spatula of <b>FA 9</b> into a dry boiling tube and add dilute hydrochloric acid until no further reaction occurs.                                                                                                | <u>Effervescence (✓) observed.</u><br><u>White residue dissolved (✓) in HCl to form colourless solution.</u><br><u>Gas evolved formed white ppt with <math>\text{Ca(OH)}_2(\text{aq})</math> / limewater (✓).</u><br><u>Gas evolved is carbon dioxide (✓).</u> |
|    | (b) To 1 cm depth of the solution from <b>test 1(a)</b> in another test tube, add aqueous sodium hydroxide dropwise until no further change is seen.<br><br><b>Keep the remaining solution from test 1(a) for 4(b)(iii).</b> | <u>White ppt (✓) formed, soluble in excess NaOH (aq) (✓) to form colourless solution.</u>                                                                                                                                                                      |

|    |                                                                                                                                                                                             |                                                                                                                                                                                                                                                                           |
|----|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 2. | To 1 cm depth of <b>FA 10</b> in a test tube, add aqueous ammonia dropwise until no further change is seen.<br><br>Note: There is no need to perform this test.                             | Blue ppt formed, soluble in excess aqueous ammonia to form a dark blue solution.                                                                                                                                                                                          |
| 3. | To another 1 cm depth of <b>FA 10</b> in a boiling tube, add a piece of aluminium foil and 2 cm depth of aqueous sodium hydroxide.<br>Heat the mixture.<br><br>Cool the mixture and filter. | Gas turned <u>damp red litmus paper blue</u> (✓). Gas evolved is <u>ammonia</u> (✓).<br><br>Blue ppt turned into <u>black / (dark) brown / grey ppt</u> (✓).<br><br><u>Black / (dark) brown / grey residue</u> (✓) and <u>blue / pale blue / colourless filtrate</u> (✓). |
| 4. | Use a glass rod to transfer the residue in <b>test 3</b> into a test tube and add 2 cm <sup>3</sup> of <b>FA 2</b> .                                                                        | <u>Black / (dark) brown / grey residue dissolved</u> (✓) to form <u>pale blue / colourless solution</u> (✓).                                                                                                                                                              |

[6]

- (b) (i) Identify the cation that is **definitely** present in **FA 8** and **two possible** identities for the other cation present in **FA 8**. [1]

Cation that is **definitely** present:  $\text{Cu}^{2+}$

**Two possible** identities for the other cation present:

$\text{Al}^{3+}$  or  $\text{Zn}^{2+}$  or  $\text{Pb}^{2+}$

- (ii) Describe a test, which will allow you to determine which of the two possible cations that you listed in 4(b)(i) is present in **FA 8**. [1]

Add 1 cm<sup>3</sup> aqueous ammonia.  $\text{Al}^{3+}$  forms white ppt that is insoluble in excess  $\text{NH}_3$  (aq) while  $\text{Zn}^{2+}$  forms white ppt that is soluble in excess  $\text{NH}_3$  (aq).

OR

[Turn Over

Add 1 cm<sup>3</sup> aqueous Na<sub>2</sub>CO<sub>3</sub>. Al<sup>3+</sup> forms effervescence while Zn<sup>2+</sup> does not form effervescence. Gas evolved formed white ppt with Ca(OH)<sub>2</sub>(aq) / limewater.

- (iii) Perform the test you describe in **4(b)(ii)** using the remaining solution [1]  
from **test 1(a)** of **Table 4.1**. Record your observations and hence deduce the other identity of the cation present in **FA 8**.

White ppt formed is soluble in excess NH<sub>3</sub> (aq) to form a colourless solution.

Cation is Zn<sup>2+</sup>.

- (iv) Given that **FA 8** does not contain nitrite ions, NO<sub>2</sub><sup>-</sup>, or sulfite ions, SO<sub>3</sub><sup>2-</sup>, identify the two anions that are present in **FA 8**. Use evidence from your observations in **4(a)** to support your deduction. [2]

Anion: CO<sub>3</sub><sup>2-</sup>

Evidence: CO<sub>3</sub><sup>2-</sup> reacts with HCl to form CO<sub>2</sub>, which gives white ppt with limewater.

Anion: NO<sub>3</sub><sup>-</sup>

Evidence: NO<sub>3</sub><sup>-</sup> reacts with NaOH and Al to give NH<sub>3</sub>, which turns damp red litmus paper blue.

- (c) (i) Write an equation to show the change in observation in **test 4**. [1]



Black residue    Blue solution

OR



- (ii) The solution prepared in **test 4** can be used to prepare a reagent to test for the carbonyl functional group.

Use this information and the observation given in **Table 4.2** to plan an experiment using the reagent prepared in **test 4** to confirm the presence of aldehyde in 1 cm<sup>3</sup> of C<sub>8</sub>H<sub>8</sub>O.

In your plan you should include brief details of:

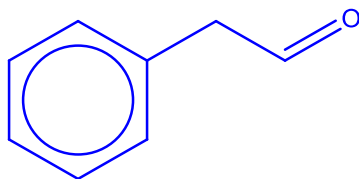
- the quantity and the identity of the reagent to be used
- the condition and apparatus you would use

**Table 4.2**

| Test                                                                                                                                                                              | Observation              |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------|
| To 1 cm <sup>3</sup> of C <sub>8</sub> H <sub>8</sub> O in a test-tube, add<br>1 – 2 cm <sup>3</sup> of Fehling's solution.<br>Warm the mixture in a water bath for<br>3 – 5 min. | Brick red ppt is formed. |

[1]

- (iii) A student carried out the plan in 4(c)(ii) and obtained the expected observation to confirm the functional group in C<sub>8</sub>H<sub>8</sub>O. Draw the structure of C<sub>8</sub>H<sub>8</sub>O. [1]



[Total: 14]

[Turn Over]

**Qualitative Analysis Notes**

[ppt. = precipitate]

**(a) Reactions of aqueous cations**

| <b>cation</b>                                  | <b>reaction with</b>                                                                |                                                                                     |
|------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
|                                                | NaOH(aq)                                                                            | NH <sub>3</sub> (aq)                                                                |
| aluminium,<br>Al <sup>3+</sup> (aq)            | white ppt.<br>soluble in excess                                                     | white ppt.<br>insoluble in excess                                                   |
| ammonium,<br>NH <sub>4</sub> <sup>+</sup> (aq) | ammonia produced on heating                                                         | –                                                                                   |
| barium,<br>Ba <sup>2+</sup> (aq)               | no ppt. (if reagents are pure)                                                      | no ppt.                                                                             |
| calcium,<br>Ca <sup>2+</sup> (aq)              | white ppt. with high [Ca <sup>2+</sup> (aq)]                                        | no ppt.                                                                             |
| chromium(III),<br>Cr <sup>3+</sup> (aq)        | grey-green ppt.<br>soluble in excess<br>giving dark green solution                  | grey-green ppt.<br>insoluble in excess                                              |
| copper(II),<br>Cu <sup>2+</sup> (aq)           | pale blue ppt.<br>insoluble in excess                                               | blue ppt.<br>soluble in excess<br>giving dark blue solution                         |
| iron(II),<br>Fe <sup>2+</sup> (aq)             | green ppt., turning brown on<br>contact with air<br>insoluble in excess             | green ppt., turning brown on<br>contact with air<br>insoluble in excess             |
| iron(III),<br>Fe <sup>3+</sup> (aq)            | red-brown ppt.<br>insoluble in excess                                               | red-brown ppt.<br>insoluble in excess                                               |
| magnesium,<br>Mg <sup>2+</sup> (aq)            | white ppt.<br>insoluble in excess                                                   | white ppt.<br>insoluble in excess                                                   |
| manganese(II),<br>Mn <sup>2+</sup> (aq)        | off-white ppt., rapidly turning<br>brown on contact with air<br>insoluble in excess | off-white ppt.. rapidly turning brown<br>on contact with air<br>insoluble in excess |
| zinc,<br>Zn <sup>2+</sup> (aq)                 | white ppt.<br>soluble in excess                                                     | white ppt.<br>soluble in excess                                                     |

**(b) Reactions of anions**

| <b><i>anion</i></b>                       | <b><i>reaction</i></b>                                                                                                                                                                           |
|-------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| carbonate,<br>$\text{CO}_3^{2-}$          | $\text{CO}_2$ liberated by dilute acids                                                                                                                                                          |
| chloride,<br>$\text{Cl}^-(\text{aq})$     | gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$ )                                                                                                            |
| bromide,<br>$\text{Br}^-(\text{aq})$      | gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$ )                                                                                             |
| iodide,<br>$\text{I}^-(\text{aq})$        | gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$ )                                                                                                         |
| nitrate,<br>$\text{NO}_3^-(\text{aq})$    | $\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil                                                                                                                     |
| nitrite,<br>$\text{NO}_2^-(\text{aq})$    | $\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil;<br>$\text{NO}$ liberated by dilute acids<br>(colourless $\text{NO} \rightarrow$ (pale) brown $\text{NO}_2$ in air) |
| sulfate,<br>$\text{SO}_4^{2-}(\text{aq})$ | gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)                                                                                                      |
| sulfite,<br>$\text{SO}_3^{2-}(\text{aq})$ | $\text{SO}_2$ liberated on warming with dilute acids;<br>gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids)                                                      |

**(c) Tests for gases**

| <b><i>gas</i></b>             | <b><i>test and test result</i></b>                                               |
|-------------------------------|----------------------------------------------------------------------------------|
| ammonia, $\text{NH}_3$        | turns damp red litmus paper blue                                                 |
| carbon dioxide, $\text{CO}_2$ | gives a white ppt. with limewater<br>(ppt. dissolves with excess $\text{CO}_2$ ) |
| chlorine, $\text{Cl}_2$       | bleaches damp litmus paper                                                       |
| hydrogen, $\text{H}_2$        | “pops” with a lighted splint                                                     |
| oxygen, $\text{O}_2$          | relights a glowing splint                                                        |
| sulfur dioxide, $\text{SO}_2$ | turns aqueous acidified potassium manganate(VII) from purple to colourless       |

**(d) Colour of halogens**

| <b><i>halogen</i></b>   | <b><i>colour of element</i></b> | <b><i>colour in aqueous solution</i></b> | <b><i>colour in hexane</i></b> |
|-------------------------|---------------------------------|------------------------------------------|--------------------------------|
| chlorine, $\text{Cl}_2$ | greenish yellow gas             | pale yellow                              | pale yellow                    |
| bromine, $\text{Br}_2$  | reddish brown gas / liquid      | orange                                   | orange-red                     |
| iodine, $\text{I}_2$    | black solid / purple gas        | brown                                    | purple                         |