Name:		Shift:	
Class:		Laboratory:	

ST ANDREW'S JUNIOR COLLEGE

PRELIMINARY PRACTICAL EXAMINATION

Chemistry **29 August 2017**

Higher 2 **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** the 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.

At the end of the examination, fasten all your work securely together.

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

Planning	Practical
14	41

This paper consists of **18** printed pages including this page and quantitative analysis notes.

1 Determination of the M_r of a hydrated ethandioate salt

Calcium ethanedioate is the major component of the most common type of human kidney stones. It is one of a series of salts formed from ethanedioic acid, $H_2C_2O_4$. Another of these salts can be represented by the formula $\mathbf{X}_2C_2O_4.H_2O$, where **X** is a Group 1 metal.

Solution **Q** contains 64.5 g dm^{-3} of $\mathbf{X}_2C_2O_4.H_2O$ in deionised water. You are not provided with **Q.**

FA 1 is a diluted solution of **Q**, in which 35.70 cm^3 of **Q** was made up to 250 cm^3 with deionised water in a graduated flask.

FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.

FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

In this question, you will perform a titration. The data from this titration will be used to determine:

- the concentration of $C_2O_4^{2-}$ in **Q**,
- the M_r of $\mathbf{X}_2C_2O_4.H_2O$, and hence the identity of **X**.

(a) Titration of FA 1 against FA 2

In this titration, **FA 2** is run from the burette into the conical flask containing **FA 1** and **FA 3**. Initially, the colour of the **FA 2** will take some time to disappear.

After some **FA 2** has been added, sufficient $Mn^{2+}(aq)$ ions will be present to allow the reaction to occur faster.

The end-point is reached when a permanent pale pink colour is obtained.

(i)

1. Fill the burette with **FA 2**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 1** into the conical flask.
3. Using an appropriate measuring cylinder, transfer 50.0 cm^3 of **FA 3** to the same conical flask.
4. Heat this solution to about 65 °C.
5. Run **FA 2** from the burette into this flask until a **permanent** pale pink colour is obtained.
6. Record your titration results in the space provided on page 3. Make certain that your recorded results show the precision of your working.
7. Repeat points **1** to **6** as necessary until consistent results are obtained.
8. **Turn off your Bunsen burner**.

Results

[5]

(ii) From your titrations, obtain a suitable volume of **FA 2** to be used in your calculations.

Show clearly how you obtained this volume.

Volume of **FA 2** =…….. **[1]**

(b) **(i)** The equation for the reaction between ethanedioate ions and manganate(VII) ions is shown below.

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

Calculate the amount, in moles of ethanedioate ions, $C_2O_4^{2-}$ in 25.0 cm^3 of **FA 1**.

Amount of $C_2O_4^{2-}$ in 25 cm^3 of **FA 1** =……. **[1]**

1 **(b)** **(ii)** Determine the concentration, in mol dm^{-3}, of $C_2O_4^{2-}$ in **Q**.

Concentration of $C_2O_4^{2-}$ in **Q** =…… **[2]**

(iii) Use your answer to **(b)(ii)** to calculate the M_r of the ethanedioate salt.

M_r of the ethanedioate salt = ……..

Hence, deduce the identity of **X.**
Show your working.

[A_r: C, 12.0; O, 16.0; H, 1.0; Li, 6.9; Na, 23.0; K, 39.1; Rb, 85.5; Cs, 132.9; Fr. 223.0]

[3]

X is ……..

1 **(c)** A student performed the experiment in **(a)(i)** using a sample of another ethanedioate salt. The student obtained a mean titre value of 22.20 cm^3. The teacher calculated that the volume of **FA 2** required should have been 22.40 cm^3. The teacher told the student that the total percentage error from the apparatus in the experiment was 0.4 %.

Calculate the error in the student's result, based on these data. State **and** explain whether or not the student's result is accurate.

……

……..... **[2]**

[Total: 14]

2 Evaluation of the reliability of a gas collection method in determining a value for the M_r of the ethanedioate salt.

In this experiment, you will react the ethanedioate salt with potassium manganate(VII), in the presence of a small amount of manganese(II) ions. You will measure the volume of CO_2 gas produced at timed intervals and determine the maximum volume of CO_2 gas produced.

FA 4 is a solution containing manganese(II) ions, Mn^{2+}.

You will need access to the **FA 1**, **FA 2** and **FA 3** solutions you used earlier.

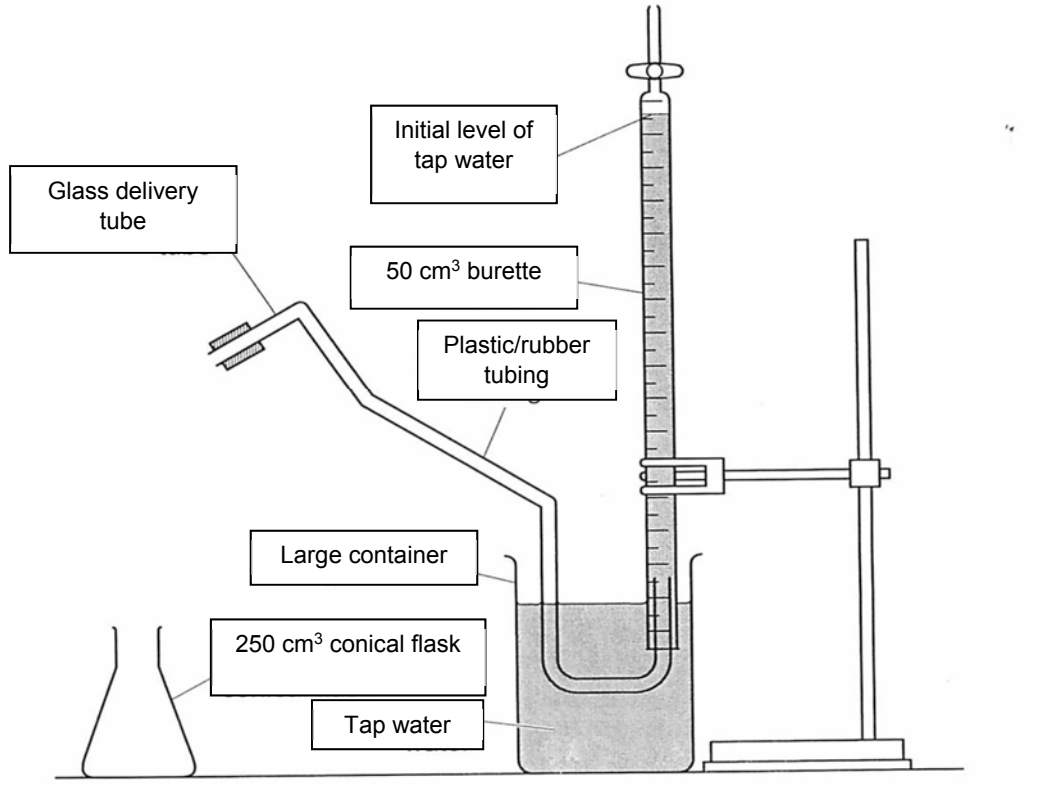


Fig. 2.1

You will measure the level of water in the burette at timed intervals.

In an appropriate format in the space provided on page 7, prepare a table in which you may record each burette reading and the time it was taken.

In addition, your table will need to show the total volume of CO_2 collected up to that time, recorded to one decimal place.

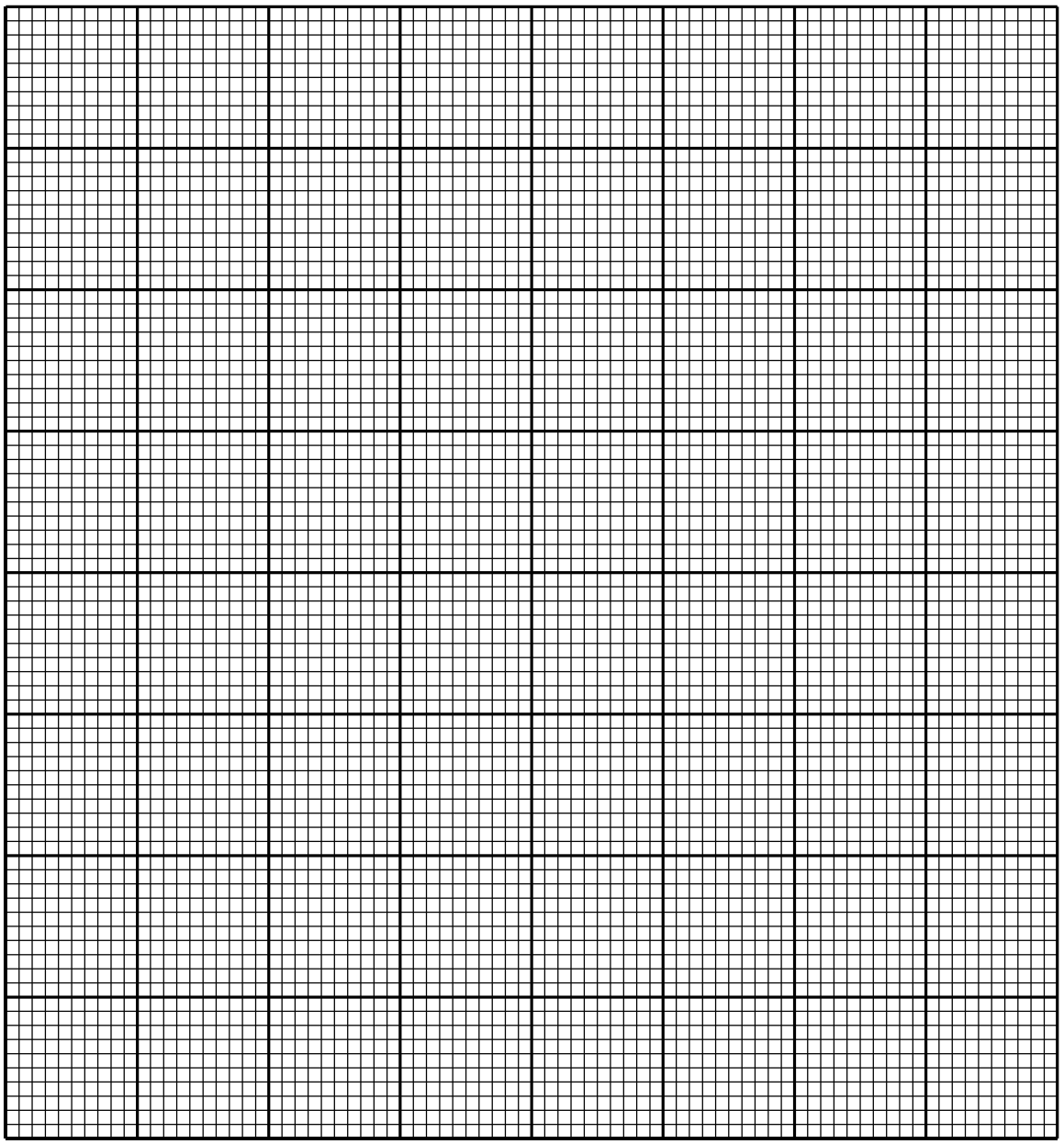
1. Set up the apparatus as shown in Fig. 2.1. You should insert the plastic/rubber tubing to a sufficient depth so that it will not subsequently shake loose.
2. Adjust the water level in the burette until it is between 48.0 cm^3 and 50.0 cm^3. You may find it helpful to use an empty dropping pipette to introduce small amounts of air from the bottom of the burette tube.
3. Use appropriate measuring cylinders to add to the 250 cm^3 conical flask.
   - 20.0 cm^3 of **FA 1**
   - 50.0 cm^3 of **FA 3**
4. Using a dropping pipette, add about 1 cm^3 of **FA 4** to the conical flask.
5. Using an appropriate measuring cylinder, measure out 30.0 cm^3 of **FA 2.**
6. Transfer the **FA 2** into the conical flask and insert the bung into the conical flask.
7. Allow any bubbles created in the burette when the bung was inserted in the conical flask to rise to the top. Start the stopwatch, read and record the initial water level in the burette.
8. Note: Once you have started the stopwatch, allow it to continue running for the duration of the experiment. You must not stop the stopwatch until the reaction is complete.
9. Check that the plastic/rubber tubing is securely positioned in the burette.
10. Hold the flask by its neck and gently swirl it continuously.
11. At t = 0.5 min, read and record the water level in the burette, to 1 decimal place, together with the time it was measured, in your table.
12. Continue to gently swirl the flask. Read and record the water level in the burette every half minute, until the reaction is complete.

(a) (i) Experimental Results

[3]

2 **(a)** **(ii)** Plot on the grid below, a graph of the volume of CO_2 on the y-axis, against time, *t,* on the x-axis.

Draw the most appropriate line, taking into account all of your points.



[4]

2 **(a)** **(iii)** The equation for the reaction between ethanedioate ions and manganate(VII) ions is shown below.

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

Use this equation, and appropriate data from your graph, to calculate a value for the amount, in moles of ethanedioate ions, $C_2O_4^{2-}$, present in 20.0 cm^3 of **FA 1**.

[molar volume of gas = 24.0 $dm^3\,mol^{-1}$ at r.t.p.]

[1]

Amount of ethanedioate ions, $C_2O_4^{2-}$, in 20.0 cm^3 of **FA 1** = ……..

(b) **(i)** Using your answer to question **1(b)(i)**, calculate the amount of ethanedioate ions in 20.0 cm^3 of **FA 1**. Hence, determine the maximum volume of CO_2 at r.t.p. that could have been produced from 20.0 cm^3 of **FA 1**.

maximum volume of CO_2 produced from 20.0 cm^3 of **FA 1** =……

[4]

(ii) Suggest a reason for the difference between the total volume of CO_2 you collected and the maximum volume of CO_2 calculated in **2(b)(i)**.

…….. **[1]**

(iii) Suggest an improvement to this experiment that would overcome this problem.

…….. **[1]**

2 **(b)** **(iv)** In Question **1** you calculated a value for the M_r of the ethanedioate salt. The total volume of CO_2 collected in **2(a)(i)** could also be used to calculate a value for the M_r of the ethanedioate salt.

Suggest which of these two *Mr* values would be higher. Explain your answer.

……

……

…… **[1]**

(c) The presence of Mn^{2+} ions, which are produced in the reaction between MnO_4^- ions and $C_2O_4^{2-}$ ions, is thought to catalyse this reaction.

(i) A student performed the experiment you performed in **2(a)(i)** but forgot to add **FA 4** to the mixture of **FA 1** and **FA 3** before adding the **FA 2**.

The student performed the experiment at the same temperature as your experiment and obtained the graph shown in **Fig. 2.2**.

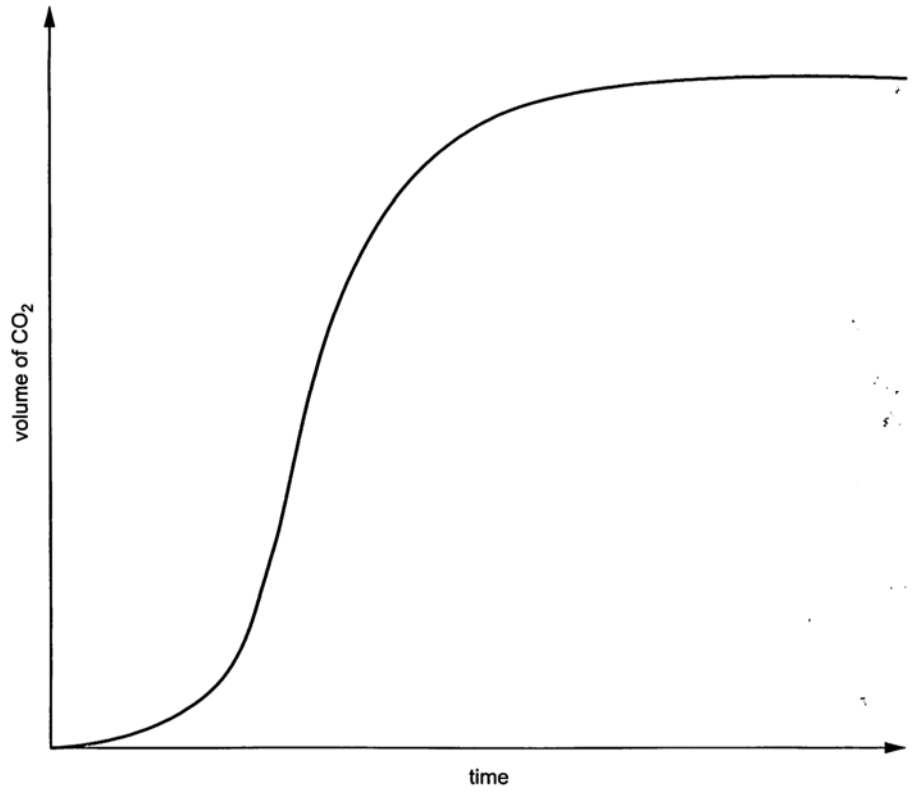


Fig. 2.2

2 (c) (i) Consider the **shape** of the graph in **Fig 2.2** and your graph in **2(a)(ii)**.
Describe one major difference between the shapes. Suggest an explanation for your answer.

Difference ……

……..

Explanation ……

……

…….. **[2]**

(ii) For the titration in **1(a)(i)** between ethanedioate ions, $C_2O_4^{2-}$, and manganate(VII) ions, MnO_4^-, the solution needs to be at about 65 °C at the start. As cold **FA 2** is added, the temperature of the mixture decreases. However, this decrease in temperature does not cause the rate of reaction between $C_2O_4^{2-}$ ions and MnO_4^- ions added from the burette to decrease.
Suggest an explanation for this.

……

……

……

…… **[1]**

2 (d) Planning

The oxidation of iodide ions, I^-, by peroxodisulfate ions, $S_2O_8^{2-}$, is known to be catalysed by Fe^{2+} or Fe^{3+} ions.

A similar reaction, shown below, in which ethanedioate ions, $C_2O_4^{2-}$, are oxidised by peroxodisulfate ions, $S_2O_8^{2-}$, may be catalysed by Cu^{2+} ions and by Ag^+ ions.

$$S_2O_8^{2-} + C_2O_4^{2-} \rightarrow 2\ SO_4^{2-} + 2\ CO_2$$

(i) Suggest an explanation why both of these reactions are slow when performed in the absence of a catalyst.

……

……

…… **[1]**

(ii) Plan an investigation to test how well, **if at all**, Cu^{2+} ions and by Ag^+ ions catalyse the reaction between $C_2O_4^{2-}$ ions and $S_2O_8^{2-}$ ions.

In your plan, you should include details of:

- the reactants and conditions that you would use,
- the apparatus you would use and the procedure you would follow,
- the measurements you would take,
- an outline of how you would use your results to compare the effectiveness of each ion as a potential catalyst.

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…… **[7]**

[Total: 26]

3 You are provided with the solid **K12** which contains one cation given in the Qualitative Analysis Notes.

You are to perform the tests below to identify the cation present in **K12** and suggest the nature of **K12.** Record your observations in the spaces provided. Your answers should include

- Details of colour changes and precipitates formed,
- The names of any gases evolved and details of the test used to identify each one.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional or confirmatory tests for ions present should be attempted.

	Tests	Observations and Deductions
1	Heat **K12** alone.	
2	Warm **K12** with dilute hydrochloric acid. Keep the solution.	
3	To the solution from test **2**, add dilute nitric acid and barium nitrate(V) solution	
4	Warm **K12** with a freshly made solution of iron(II) sulfate solution.	
5	Dissolve **K12** in dilute nitric acid. Add manganese(II) sulfate solution and two drops of silver nitrate(V) solution to act as a catalyst. Boil the mixture.	
6	Warm **K12** with sodium hydroxide solution.	

Nature of **K12:** _____

Cation it contains: _____ **[9]**

[Total: 9]

4 Planning

You are provided with 3 unlabelled bottles and each bottle contains one of the following organic compounds,

- Butanamide
- Lactic acid, $CH_3CH(OH)CO_2H$
- Pyruvic acid, CH_3COCO_2H

(a) Outline a sequence of simple chemical tests to identify each of the above organic substances. You may use any other common laboratory chemical reagents and apparatus, if necessary. You are **not** allowed to identify the substances by elimination. You are reminded that most of the compounds listed are *flammable* liquids. **[3]**

Test	**Expected Observations & Deductions**

(b) Suggest a safety measure that you would consider in carrying out your plan.

_____

_____ **[1]**

(c) State the required reagents and conditions. Draw a set-up of the apparatus for the synthesis of pyruvic acid from lactic acid.

[2]

[Total: 6]

The End