H

NANYANG JUNIOR COLLEGE
JC2 PRACTICAL PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME	

CLASS	1	7			REG NO.			TUTOR	

CHEMISTRY — 9729/04

Paper 4 Practical

28 August 2018

2 hour 30 minutes

Candidates answer on the Question Paper

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name and class on all the work you hand in.
Give details of the practical shift and laboratory, where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use an HB pencil for any diagrams or graphs
Do not use staples, paper clips, glue or correction fluid.

Answer **all** questions in the spaces provided on the Question Paper.
The use of an approved scientific calculator is expected, where appropriate.
You may lose marks if you do not show your working or if you do not use appropriate units.
Qualitative Analysis Notes are printed on pages 22 and 23.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [ ] at the end of each question or part question.

Shift
Laboratory

For Examiner's Use	
1	/ 28
2	/ 11
3	/16
Total	/55

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

1 **To determine the concentrations of aqueous sodium hydroxide and aqueous ethanedioic acid and hence the percentage by mass of sodium ethanedioate in a mixture of sodium ethanedioate and ethanedioic acid.**

You are provided with the following reagents.

- **FB 1** is a mixture of approximately 0.01 mol dm^{-3} aqueous sodium ethanedioate, $C_2O_4Na_2$, and approximately 0.1 mol dm^{-3} ethanedioic acid, $C_2O_4H_2$
- **FB 2** is aqueous sodium hydroxide, NaOH
- **FB 3** is 0.0755 mol dm^{-3} aqueous sodium carbonate
- thymolphthalein indicator

Both sodium hydroxide and sodium carbonate are bases which will neutralise ethanedioic acid as shown in **reactions 1** and **2**.

$2NaOH + C_2O_4H_2 \rightarrow C_2O_4Na_2 + 2H_2O$ **reaction 1**

$Na_2CO_3 + C_2O_4H_2 \rightarrow C_2O_4Na_2 + CO_2 + H_2O$ **reaction 2**

In this experiment, you will add different volumes of **FB 3** to identical samples of the mixture of aqueous sodium ethanedioate, $C_2O_4Na_2$, and ethanedioic acid, $C_2O_4H_2$, **FB 1**. You will then complete the neutralisation of $C_2O_4H_2$ in each mixture by titrating with dilute sodium hydroxide, **FB 2**.

Each titration is to be performed **once only**, so great care should be taken that you do not exceed the end-points.

Graphical analysis of your results will enable you to determine the concentrations of the ethanedioic acid in the mixture of aqueous $C_2O_4Na_2$, and $C_2O_4H_2$, **FB 1** and of the sodium hydroxide, **FB 2**.

Read through the whole method of conducting the experiment before starting any practical work.

The experiment

(a) Titrations

Experiment 1

1. Fill a burette with **FB 3**.
2. Pipette 25.0 cm^3 of **FB 1** into a 250 cm^3 conical flask.
3. Run 5.00 cm^3 of **FB 3** into the flask.
4. Add 10 – 15 drops of thymolphthalein indicator to the flask.
5. Fill a second burette with **FB 2**.
6. Titrate the mixture in the conical flask against **FB 2** until the first permanent pale blue colour remains in the solution.
7. Rinse the conical flask thoroughly before it is used for the next experiment.

The end-point should be found after the addition of approximately 14 cm^3 of **FB 2**.

One titration, performed accurately, will be sufficient.

Experiment 5

1. Repeat **Experiment 1** but run 30.00 cm^3 of **FB 3** into the flask containing 25.0 cm^3 of **FB 1**.

Experiments 2 – 4

Select **three** other suitable volumes of **FB 3** for use in **Experiments 2 – 4**. Your selected volumes must be between the volumes used in **Experiments 1 and 5**.

Do not use volumes of FB 3 outside the range of 5 – 30 cm^3.

Repeat the procedures used in **Experiment 1** but, in each case, add 25.0 cm^3 of **FB 1** and your chosen volume of **FB 3** into the conical flask. You should perform your titrations in order of **increasing** volume of **FB 3**.

Rinse this conical flask thoroughly between each titration.

Prepare a table in the space below and use it to record the titration results for each volume of **FB 3** added. You should record your titration results in order of **increasing** volume of **FB 3** added.

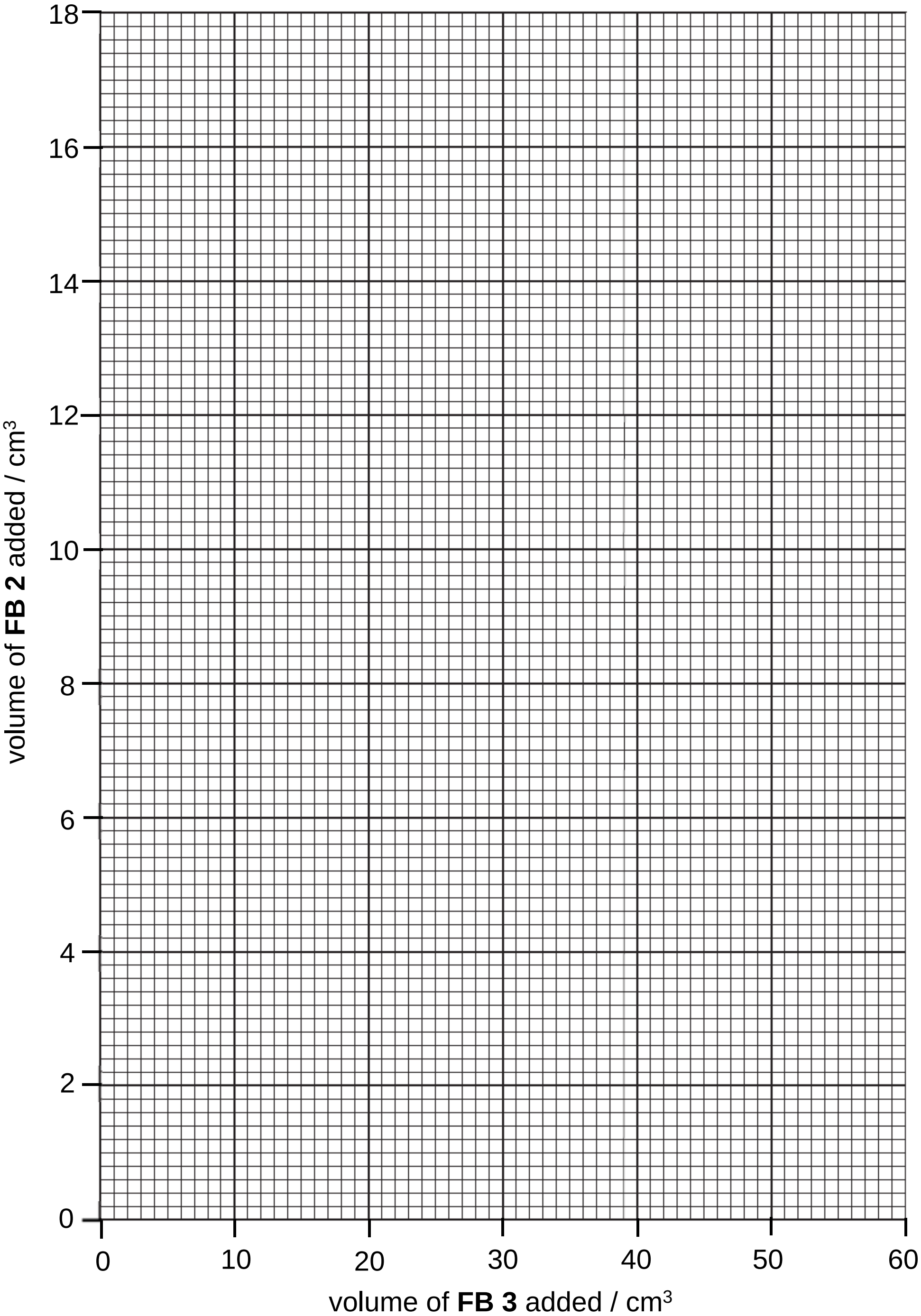
I	
II	

[2]

(b) **(i)** Plot, on the grid below, your values for the **FB 2** titre (*y-axis*) against the volume of **FB 3** added (*x-axis*).

Draw the line of best fit, taking into account all of your plotted points. Hence obtain values for

- the volume of **FB 3** required, $\mathbf{V_{max}}$**(FB 3)**, to react completely with 25.0 cm^3 of **FB 1** if no **FB 2** is added;
- the volume of **FB 2** required, $\mathbf{V_{max}}$**(FB 2)**, to react completely with 25.0 cm^3 of **FB 1** if no **FB 3** is added.



Teacher's V_{max}**(FB 3)**	
diff.	
Teacher's V_{max}**(FB 2)**	
diff.	
I	
II	
III	
IV	
V	

$\mathbf{V_{max}}$**(FB 3)** = ……………….. $\mathbf{V_{max}}$**(FB 2)** = ………………..

[5]

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

gradient = ... [1]

(iii) Explain, in terms of the chemistry involved, the direction of the slope of your graph.

....

....

....

.... [1]

Calculations
Show your working and appropriate significant figures in **all** of your calculations.

(c) **(i)** Using appropriate data from your graph, calculate the concentration of ethanedioic acid, $C_2O_4H_2$ in **FB 1**.

concentration of $C_2O_4H_2$ in **FB 1** = ... [1]

(ii) Using your answer to **(c)(i)** and appropriate data from your graph, calculate the concentration of sodium hydroxide in **FB 2**.

concentration of sodium hydroxide in **FB 2** = ... [1]

(d) Use the expression below to calculate the concentration of sodium hydroxide in **FB 2**.

$$[NaOH] = \frac{2[Na_2CO_3]}{|\,gradient\,|}$$

concentration of sodium hydroxide in **FB 2** = ... [1]

(e) Student **B** repeats the experiment described in **(a)**. However, student **A** had used up **FB 3**. As such student **B** has to prepare another 250 cm^3 of **FB 3**. Unknowingly, he weighs a sample of solid sodium carbonate which is slightly damp for the preparation of **FB 3**.

Suggest and explain what effect this will have on the value of $\mathbf{V_{max}}$**(FB 3)** he obtains.

effect on $\mathbf{V_{max}}$**(FB 3)** ………………………………………………………………..

explanation ……………………………………………………………………………

……………………………………………………………………………………………

………………………………………………………………………………………… [1]

(f) A student performs the original experiment using solutions of different concentration to those you have used. He determines the gradient of his line using a small triangle.

He calculates the concentration of NaOH in **FB 2**. In part **(c)(ii)** he obtains a value of 0.589 mol dm^{-3}. In part **(d)** he obtains a value of 0.581 mol dm^{-3}.

Suggest which of these two values is likely to be more accurate. Explain your answer.

the more accurate value is ………………………………………………………………..

explanation ……………………………………………………………………………

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………………………………………………………………………………………… [1]

(g) Plan an experiment to determine the percentage by mass of sodium ethanedioate, $C_2O_4Na_2$ in **FB 1**.

Acidified potassium manganate(VII) oxidises ethanedioate ions, $C_2O_4^{2-}$ ions, as shown below.

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

However, the reaction takes place slowly at first. The rate of the reaction increases as more products are produced. This is because one of the products acts as a catalyst for the reaction. Hence this reaction is an example of 'autocatalysis'.

(i) Identify the product that acts as the catalyst in the reaction.

………………………………………………………………………………… [1]

(ii) Using the information given above, complete the procedures for this additional experiment which is written partially as shown below.

*Do **NOT** carry out this experiment.*

1. Fill a burette with 0.020 mol dm^{-3} of an aqueous solution of potassium manganate(VII).
2. Pipette 10.0 cm^3 of **FB 1** into a 250 cm^3 conical flask.
3. Using a 10 cm^3 measuring cylinder, add in 10 cm^3 of sulfuric acid into the same conical flask.
4. …………………………………………………………………………………

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   …………………………………………………………………………………
5. Titrate the mixture in the conical flask against potassium manganate(VII) until the first permanent ……………………… colour remains in the solution.
6. Repeat the titration ……………………………………………………………

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[2]

(h) Planning

The volume of aqueous sodium hydroxide needed to completely neutralise all the ethanedioic acid, $C_2O_4H_2$ present in **FB 1** can also be obtained by carrying out a thermometric titration.

The reaction between an acid and an alkali is exothermic. It is possible to make use of this fact to determine the equivalence-point of a neutralisation reaction without the use of an indicator. This process is known as *thermometric titration* and can be used to calculate the concentration of an acid solution and the value of the enthalpy change of neutralisation, ΔH_n.

The concentration of an acid solution can be determined by adding various volumes of acid and alkali and measuring the change in temperature. A series of about six experiments were conducted, where different volumes of alkali were added to a fixed volume (for example 25.0 cm^3) of acid while keeping the total volume of the solution constant by adding appropriate volumes of water.

The data obtained is plotted and two best-fit graph lines are drawn. One line is drawn using data before the equivalence-point and the second line using the remaining data. These lines are then extrapolated (extended) until they cross.

(i) Using the information given above, you are required to write a plan for a thermometric titration in which a known concentration of an aqueous solution of sodium hydroxide is added to **FB 1**.

You are to determine the volume of aqueous sodium hydroxide required to completely neutralise the ethanedioic acid present in **FB 1**.

You may also assume that you are provided with:

- **FB 1** which is a mixture of approximately 0.01 mol dm^{-3} aqueous sodium ethanedioate, $C_2O_4Na_2$, and approximately 0.1 mol dm^{-3} ethanedioic acid, $C_2O_4H_2$.
- 0.35 mol dm^{-3} aqueous sodium hydroxide, NaOH
- graph paper;
- the equipment normally found in a school or college laboratory.

Your plan should include:

- calculation of the approximate volume of aqueous sodium hydroxide required to completely neutralise the ethanedioic acid present in 25.0 cm^3 of **FB 1**;
- brief, but specific, details of the apparatus you would use, bearing in mind the levels of precision they offer;
- an outline of how the results would be obtained;
- a table containing the volumes of each reagent to be added
- how you would recognise that the equivalence-point had been passed;
- on the grid provided, a sketch of the graph you would expect to obtain;
- an explanation of the shape of your graph;

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Explanation of the shape of the graph:

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……………………………………………………………………………..……………. [8]

(ii) In another experiment, hydrochloric acid is used instead of **FB 1**.
If the hydrochloric acid has the same concentration as the ethanedioic acid present in **FB 1**, draw on your graph in **(i)** another pair of lines to show the results you would expect to obtain.

Explain your answer.

……………………………………………………………………………………….

……………………………………………………………………………………….

……………………………………………………………………………………….

……………………………………………………………………………………….

……………………………………………………………………………………. [3]

[Total: 28]

2 **You are required to determine the enthalpy change of combustion of ethanol.**

When an exothermic reaction takes place in a container such as a metal calorimeter, some of the evolved heat energy is absorbed by the metal calorimeter.

When an endothermic reaction takes place some of the required heat energy is supplied by the metal calorimeter.

The amount of heat energy evolved or supplied for a 1 °C change in temperature is known as the heat capacity of the metal calorimeter.

In preparation for your experiment to determine the enthalpy change of combustion of ethanol, you will first need to determine the approximate heat capacity of a 100 cm^3 metal calorimeter.

Before starting any practical work read through the instructions in (a) and draw up a table to record your results.

(a) **Determining the approximate heat capacity of the 100 cm^3 metal calorimeter**

When samples of hot and cold water are mixed in the 100 cm^3 metal calorimeter, some heat is lost to the metal calorimeter in raising its temperature. To determine the approximate heat capacity of your 100 cm^3 metal calorimeter, you will determine the maximum temperature rise when a sample of hot water is added to cold water in the metal calorimeter.

- Use a 50 cm^3 measuring cylinder to transfer 50 cm^3 of cold water into the 100 cm^3 metal calorimeter.
- Use the 50 cm^3 measuring cylinder to transfer 50 cm^3 of cold water into a 100 cm^3 beaker.
- Note the temperature of the water in this 100 cm^3 beaker and heat it **carefully and gently** until the temperature of the water in it has increased by 45–50 °C then stop heating, *e.g. if the water is at 20.0 °C you should warm it to 65–70 °C.*
- Stir the cold water in the 100 cm^3 metal calorimeter with the thermometer.
- Record the temperature of the cold water (this is the temperature at t = 0 min).
- Record the temperature each minute for 3 minutes.
- After you have taken the reading at t = 3 min, use the thermometer to stir the hot water in the 100 cm^3 beaker.
- At t = 4 min, measure the temperature of the hot water and record this value in the box below.
- **Immediately** add the hot water from the 100 cm^3 beaker to the cold water in the 100 cm^3 metal calorimeter.
- Stir with the thermometer but do not record the temperature.
- Continue to stir the water throughout the experiment.
- Record the temperature at t = 5 min, and then every ½ minute until t = 8 min.
- Record all measurements of time and temperature obtained on page 13.

The temperature, $\boldsymbol{T_1}$, of the hot water at t = 4 min is …………………. °C.

Table of results

I	
II	
III	

[3]

(b) Graph plotting

1. Plot a graph of the temperature of the water in the 100 cm^3 metal calorimeter (y-axis) against time (x-axis) on the grid below. Do not plot the temperature, $\boldsymbol{T_1}$, of the hot water at t = 4 min.
2. Draw two straight lines of best fit; one through the points up to t = 3 min; the second through the points from t = 5 min to t = 8 min. Extrapolate both lines to t = 4 min.
3. From the extrapolated lines read the minimum and the maximum temperatures at t = 4 min. Record these values in the spaces provided below.
4. Determine the values for the two temperature changes at t = 4 min.

I	
II	
III	
IV	

Minimum temperature, $\boldsymbol{T_2}$, at t = 4 min is …………............... °C.

Maximum temperature, $\boldsymbol{T_3}$, at t = 4 min is …………............... °C.

Temperature rise for 50 cm^3 of cold water in the 100 cm^3 metal calorimeter, ($\boldsymbol{T_3} - \boldsymbol{T_2}$) is ……….…...... °C.

Temperature fall for 50 cm^3 of hot water from the 100 cm^3 beaker, ($\boldsymbol{T_1} - \boldsymbol{T_3}$) is ……….…...... °C.

[4]

(c) **Calculations**
Working should be shown in all calculations.
[**4.2 J** are absorbed or released when the temperature of 1.0 cm^3 of **water** changes by 1.0 °C.]

(i) Calculate the heat energy gained by the 50 cm^3 of cold water in the 100 cm^3 metal calorimeter.

The heat energy gained by the cold water = ………………… J.

(ii) Calculate the heat energy lost by the 50 cm^3 of hot water from the 100 cm^3 beaker.

The heat energy lost by the hot water = ………………… J.

(iii) The difference between the values calculated in **(i)** and **(ii)** is an approximate value for the total heat energy absorbed by the 100 cm^3 metal calorimeter during the experiment. The heat capacity of the metal calorimeter is the amount of heat energy absorbed for a 1 °C change in temperature.

$$\begin{array}{c}\textit{approximate heat capacity} \\ \textit{of the } 100\ cm^3 \textit{metal calorimeter}\end{array} = \frac{(\textit{heat energy lost}) - (\textit{heat energy gained})}{(T_3 - T_2)}\ \text{J °C}^{-1}$$

Use your answers to **(i)** and **(ii)** and the temperature rise from **(b)** to calculate the approximate heat capacity of the 100 cm^3 metal calorimeter.

The approximate heat capacity of the 100 cm^3 metal calorimeter = ………………… J °C^{-1}.
[1]

(d) Determining the enthalpy change of combustion of ethanol, C_2H_5OH

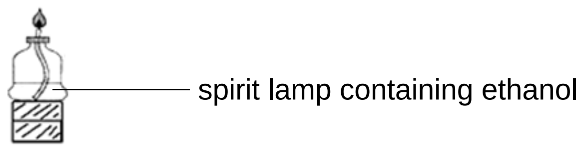
An experiment was carried out as follows to determine the enthalpy change of combustion of ethanol.

The 100 cm^3 metal calorimeter used in **(a)** was used to contain some water. The water was heated using ethanol placed in a spirit lamp. The temperature rise was recorded. The spirit lamp was weighed before and after the experiment to determine the mass of ethanol used. The following results were obtained.

mass of ethanol burned = 0.391 g
mass of water heated = 40.0 g
temperature rise = 19.5 ^oC

(i) Complete the following diagram to show the experimental set-up needed to carry out the experiment to determine the enthalpy change of combustion of ethanol.
You are to use the following apparatus to complete the diagram:

- stand and clamp
- 100 cm^3 metal calorimeter
- thermometer
- tripod stand and heat-proof mat



[1]

(ii) Use your answer in **(c)(iii)** and the results obtained to calculate the enthalpy change of combustion of ethanol.
[*A*r: O, 16.0; C, 12.0; H, 1.0]
[specific heat capacity of water = 4.2 J $^{o}C^{-1}$ cm^{-3}]
(If you were unable to answer **(c)(iii)**, you may assume that the heat capacity of the 100 cm^3 metal calorimeter is 50 J $^{o}C^{-1}$.)

enthalpy change of combustion of ethanol = ……………………..[1]

(iii) Calculate the maximum percentage error in the measurement of each mass used in the experiment.

mass measured	maximum error in a single reading	maximum percentage error / %
0.391 g of ethanol burned	0.0005 g	
40.0 g of water	0.05 g	

[1]

[Total: 11]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.
No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

FB 4 is a solution containing **one** cation and **one** anion from the Qualitative Analysis Notes in pages 22 and 23.
Solid **FB 5** contains the sodium ion and **one** anion from the Qualitative Analysis Notes in pages 22 and 23.

(a) Carry out the following tests on **FB 4** and state the observations you make.

	Test	*Observations*
(i)	To 1 cm depth of aqueous **FB 4** in a test-tube, add aqueous ammonia, then	
	add sulfuric acid dropwise until there is no further observations.	
(ii)	To 1 cm depth of aqueous **FB 4** in a test-tube, add silver nitrate,	
	followed by aqueous ammonia.	

For Examiner's Use			
Obs points		*marks*	

(b) Carry out the following tests on **FB 5** and state the observations you make.

	Test	*Observations*
(i)	Place half of the solid **FB 5** in a boiling tube. Heat strongly until the solid melts and a gas is given off. Test and identify the gas. Continue the strong heating for 2-3 min, then leave the tube to cool and retain the residue for the test in **(iv)**.	
(ii)	To the remaining half of **FB 5** in the boiling tube, add 3 cm depth of aqueous sodium hydroxide. Warm gently and retain for the test in **(iii)**.	
(iii)	To the solution from the test in **(ii)**, add a piece of aluminium foil and warm gently.	
(iv)	Dissolve the residue from test in **(i)** in distilled water and divide the solution into **three** parts. Use these for the tests in **(v)** to **(vii)**.	
(v)	To one part of the solution from **(iv)**, add aqueous potassium iodide followed by dilute sulfuric acid.	
(vi)	To the second part of the solution from **(iv)**, add aqueous potassium manganate (VII) followed by dilute sulfuric acid.	
(vii)	To the third part of the solution **(iv)**, add aqueous sodium hydroxide and a piece of aluminium foil. Warm gently.	

[6]

(c) **(i)** Suggest the identities of the cation and anion present in **FB 4** from your observations from the tests in **(a).**

cation: …………………… **anion:** …………………… [1]

(ii) Write equations, with state symbols to explain your observations for test in **a(i).**

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……………………………………………………………………………… [3]

(d) **(i)** Suggest the identity of the anion present in **FB5** ***before*** it was heated.

……………………………………………………………………………… [1]

(ii) Suggest the identity of the anion present in **FB5** ***after*** it was heated.

……………………………………………………………………………… [1]

(iii) In **(b)(v)**, the anion is behaving as ………………………….. [1]

(iv) In **(b)(vi)**, the anion is behaving as …………………………. [1]

(v) Suggest the purpose of carrying out test **(b)(ii)**.

…………………………………………………………………………………

…………………………………………………………………………………

……………………………………………………………………………… [1]

(e) Student **C** carried out the following experiment on **FB 5**, predict the observations that will be observed and write your answer in the blanks.

Test	*Observations*
To solid **FB 5** in a boiling tube. Heat heat strongly for 4 min. Let the residue cool. Add dilute sulfuric acid to it.	

[1]

[Total: 16]

Qualitative Analysis Notes
[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	***reaction with***	
	NaOH(aq)	NH_3(aq)
aluminium, Al^{3+}(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH_4^+(aq)	ammonia produced on heating	–
barium, Ba^{2+}(aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca^{2+}(aq)	white ppt. with high [Ca^{2+}(aq)]	no ppt.
chromium(III), Cr^{3+}(aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu^{2+}(aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe^{2+}(aq)	green ppt., turning brown on contact with air insoluble in excess	green ppt., turning brown on contact with air insoluble in excess
iron(III), Fe^{3+}(aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg^{2+}(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn^{2+}(aq)	off-white ppt., rapidly turning brown on contact with air insoluble in excess	off-white ppt., rapidly turning brown on contact with air insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

(b) Reactions of anions

anion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, C*l* (aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br (aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I (aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq))
nitrate, NO_3 (aq)	NH_3 liberated on heating with OH (aq) and A*l* foil
nitrite, NO_2 (aq)	NH_3 liberated on heating with OH (aq) and A*l* foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acids)
sulfite, SO_3^{2-}(aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in dilute strong acids)

(c) Tests for gases

gas	*test and test result*
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	"pops" with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

halogen	*colour of element*	*colour in aqueous solution*	*colour in hexane*
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br_2	reddish brown gas / liquid	orange	orange-red
iodine, I	black solid / purple gas	brown	purple