H

NANYANG JUNIOR COLLEGE
JC2 PRACTICAL PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME **Mark Scheme**

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

This document consists of **23** printed pages and **1** blank page.

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 a dilute solution of 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 titration with dilute sodium hydroxide, **FB 2**.

Each titration is to be performed **once only**, so great care should be taken that you do not overshoot 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 $C_2O_4H_2$ and of the sodium hydroxide.

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.

Experiment	1	2	3	4	5
Volume of **FB 3** added / cm^3	5.00	11.00	18.00	24.00	30.00
Initial burette reading / cm^3	0.00	20.00	0.00	11.70	21.60
Final burette reading / cm^3	15.80	34.00	11.70	21.60	29.80
Volume of **FB 2** added / cm^3	15.80	14.00	11.70	9.90	8.20

[1]: Correct column or row headings and units for titration recordings
Acceptable forms of units:
Use of solidus, e.g. / cm^3
Unit in brackets, e.g. (cm^3)
In words, e.g. volume in cubic centimetres
AND
All burette readings and/or volumes/titres recorded to 2 dp or to nearest 0.05 cm^3)

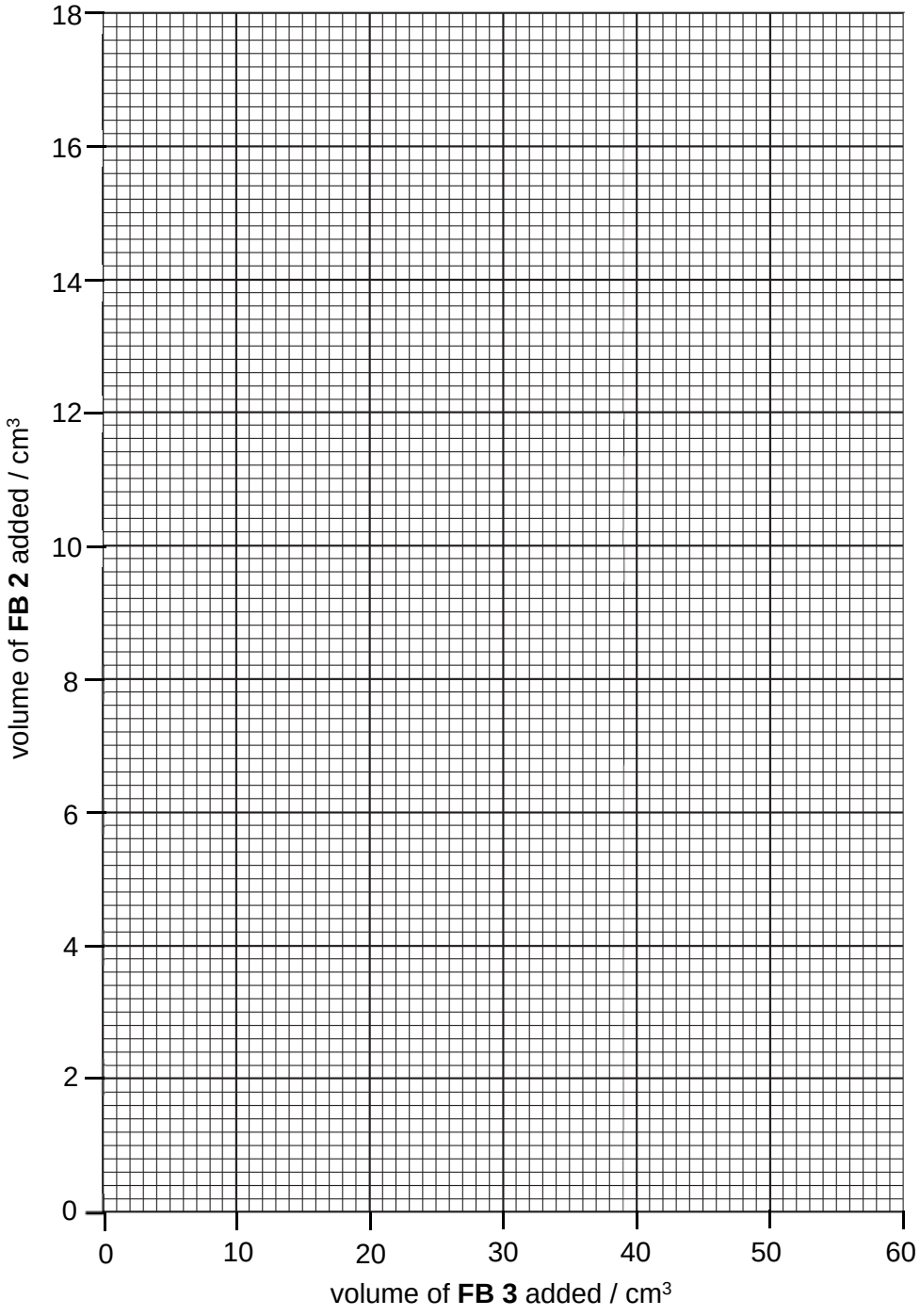
[1]: Follows instructions.
Selects three additional volumes of **FB 3** which are within the range 5 – 30 cm^3 for experiments 2, 3, and 4.
AND All gaps between adjacent **FB 3** volumes should be ≥ 5 cm^3 and ≤ 10 cm^3
AND Completes a total of 5 experiments.

[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	

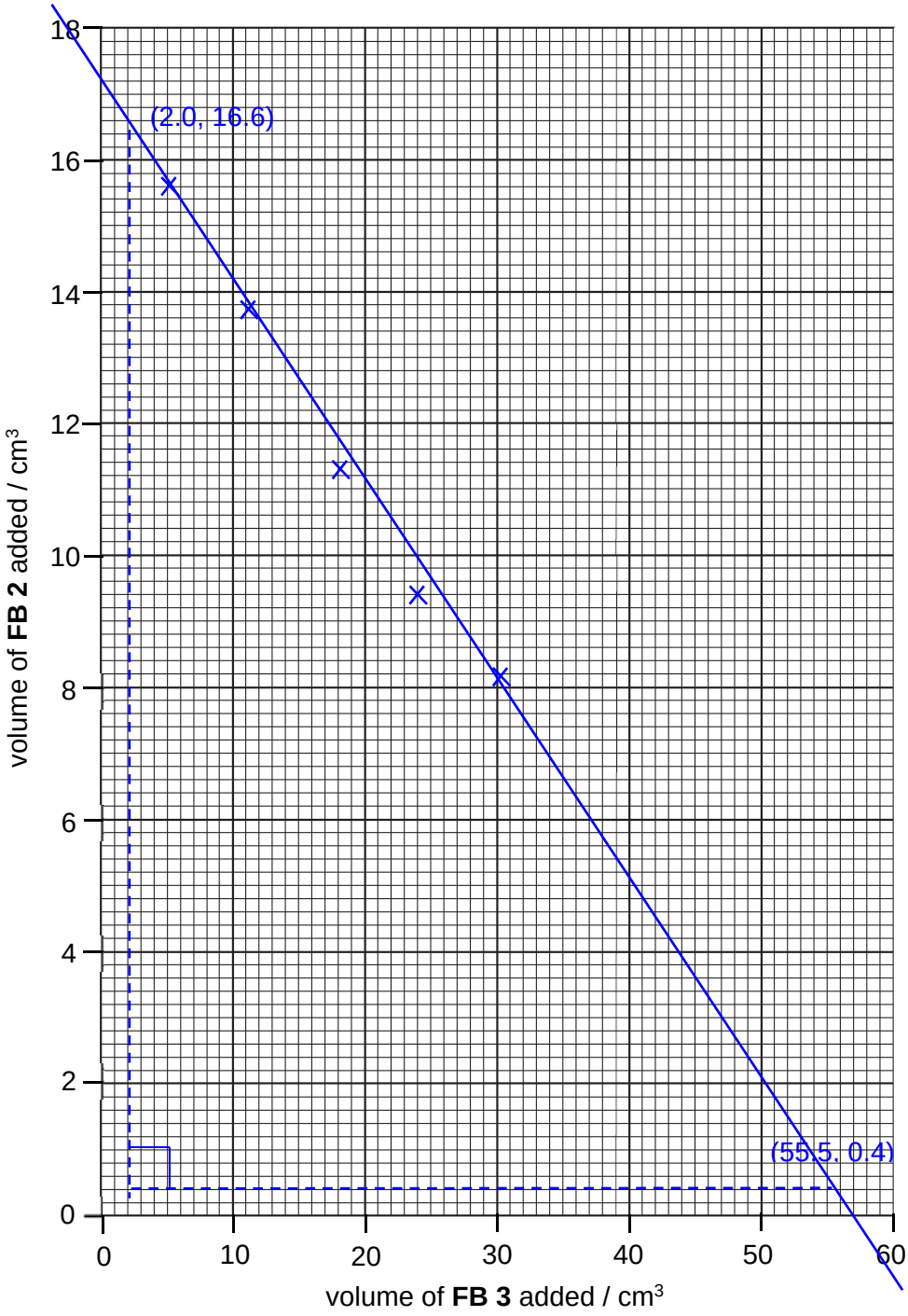
$\mathbf{V_{max}(FB\ 3)}$ = ……. $\mathbf{V_{max}(FB\ 2)}$ = …….

[5]

(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	

$\mathbf{V_{max}(FB\ 3)}$ = 57.00 cm^3…… $\mathbf{V_{max}(FB\ 2)}$ = 17.20 cm^3……

[5]

[1]: All points plotted to within ½ small square in either direction and in the correct square.

[1]: The graph line is the straight best-fit line
AND
Line is correctly extrapolated to intersect both axes. i.e. one continuous straight line
AND
no point is further than 1 cm^3 away from the line in either direction

[2]: Accuracy for V_{max}(FB 3) and V_{max}(FB 2)

[1] Reads correctly, to ±½ small square, the value for **V_{max}(FB 3)** and the value for **V_{max}(FB 2)**
If the student has not shown the extrapolation on the graph, the student may still earn this mark if the values quoted correspond to correct extrapolation of the student's graph line.

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

$$\text{gradient} = \frac{16.6 - 0.4}{2 - 55.5} = -0.3028 \approx -0.303$$

gradient = –0.303…… [1]

To earn this mark, students must
- Use clearly stated co-ordinates from graph **or** correct values of Δ*titre* and of Δ*(volume of* ***FB 3*** *used)* obtained from a clearly drawn triangle *(allow measurements to ± ½ small square). Coordinates could be from* V_{max} *values*.
- Use a triangle (or equivalent coordinates/Δ values) which covers at least 3 large squares in each direction
- Correctly calculate gradient. (ignore sig figs for this mark)

Do not award this mark if the graph line is not straight.
For this mark, ignore missing working and incorrect/missing units.

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

……

……

……

…… [1]

[1]: Negative gradient because the more Na_2CO_3 added, the less NaOH is needed for neutralisation (or words to that effect)

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**.

1 mol of Na_2CO_3 will completely neutralise 1 mol of $C_2O_4H_2$

V_{max}(**FB 3**) = 57.00 cm^3

$$[C_2O_4H_2] = \frac{57.00/1000 \times 0.0755}{25.0/1000} = 0.1721 \approx 0.172 \text{ mol dm}^{-3}$$

concentration of $C_2O_4H_2$ in **FB 1** = 0.172 mol dm^{-3} ..... [1]

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

2 mol of NaOH will completely neutralise 1 mol of $C_2O_4H_2$
V_{max}(**FB 2**) = 17.20 cm^3

$$[NaOH] = \frac{57.00/1000 \times 0.0755 \times 2}{17.20/1000} = 0.5004 \approx 0.500 \text{ mol dm}^{-3}$$

concentration of sodium hydroxide in **FB 2** = 0.500 mol dm^{-3} ... [1]

(d) Use the expression below to calculate the concentration of sodium hydroxide in **FB 2**. Give your answer to three significant figures.

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

$$[NaOH] = \frac{2(0.0755)}{0.3028} = 0.4986 \approx 0.499 \text{ mol dm}^{-3}$$

concentration of sodium hydroxide in **FB 2** = 0.499 mol dm^{-3}..... [1]

[1 mark]
Correctly calculates [NaOH] using the given expression
Show working in **(c)(ii)**, **(d)** and **(e)**
3 significant figures in final section answers to **(c)(ii)**, **(d)** and **(e)**
Show appropriate units in answers in **(c)(i)**, **(c)(ii)**, **(d)(ii)**, **(d)(iii)** and **(e)**

(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 **V_{max}(FB 3)** he obtains.

effect on **V_{max}(FB 3)** .....
value of **V_{max}(FB 3)** too high

explanation .....

.....

..... [1]

presence of water means mass of Na_2CO_3 weighed out is lower than expected, so [Na_2CO_3] in **FB 3** is lower than expected, so more **FB 3** is needed to neutralise the ethanedioic acid in **FB 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 0.589 ……..

explanation ……

……

…… [1]

because it is based on larger/the maximum/the intercept volumes.
AND
so the effect of a reading error will be less significant.

(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. ……

   ……

   ……

   ……

   Heat the mixture in the conical flask to about 60/70/80 °C.
5. Titrate the mixture in the conical flask against potassium manganate(VII) until the first permanent pale pink…….. colour remains in the solution.
6. Repeat the titration ……

   ……

   ……

   ……

   Repeat the titration as many times necessary to achieve two consistent readings (to be within ±0.10 cm^3. The average of the two consistent readings will be the volume of potassium manganate(VII) added to achieve endpoint.)

[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 measure 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**.

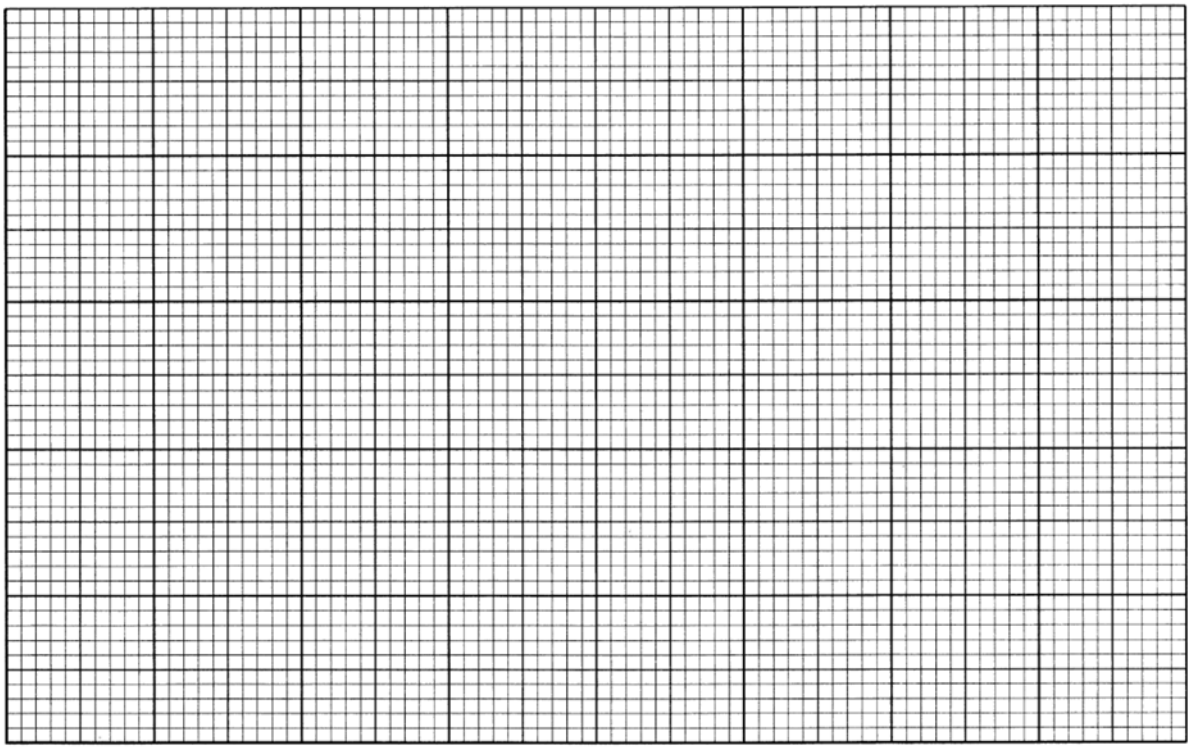
You may also assume that you are provided with:

- **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} 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 **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;

……



…… [8]

[1] Calculation:

Given approx. $[C_2O_4H_2]$ = 0.1 mol dm^{-3} and [NaOH] = 0.35 mol dm^{-3}

Given fix acid volume at 25.0 cm^3

$n(C_2O_4H_2) = \frac{25.0}{1000} \times 0.1 = 0.0025$ mol

$n(NaOH) = 2n(C_2O_4H_2) = 0.0025 \times 2 = 0.005$ mol

approximate $\mathbf{V_{max}}$**(FB 2)** = 0.005 / 0.35 × 1000 = 14.29 cm^3 (ignore the precision when presenting this answer)

Procedure:

1. Use a burette to transfer 25.00 cm^3 of **FB 1** into a styrofoam cup. (Place the cup in a 250 cm^3 beaker to prevent it tipping over.) Record the initial temperature of **FB 1** using a thermometer graduated in 0.2 ^oC.
2. From a second burette, measure the required volume of water as shown in the table below and add to the **FB 1** in the styrofoam cup. (Use the thermometer to stir the mixture gently.)
3. From a third burette, measure the required volume of NaOH as shown in the table below into a 100 cm^3 beaker and add to **FB 1** in the styrofoam cup. Use the thermometer to stir the mixture gently.
4. Record the highest temperature reached.
5. Repeat steps **1** to **4** by varying the volume of water and NaOH added to **FB 1** as shown in the table below.
6. The temperature change, $\Delta T = T_{final} - T_{initial}$, where T_{final} is the highest temperature measured and $T_{initial}$ is the initial temperature of **FB 1**.
7. Plot a graph of the ΔT against the volume of NaOH added.
8. The equivalence-point is reached once the temperature is observed to stop rising.

Experiment	Volume of NaOH / cm^3	Volume of **FB 1** / cm^3	Volume of water / cm^3	Initial temperature of **FB 1** / ^oC	Highest temperature / ^oC	ΔT / ^oC
1	8.00	25.00	22.00			
2	12.00	25.00	18.00			
3	14.00	25.00	16.00			
4	16.00	25.00	14.00			
5	18.00	25.00	12.00			
6	20.00	25.00	10.00			

Marking Scheme

1 mark for describing the followings in the procedure:

- state the use of burette to measure the volumes of water, NaOH and **FB 1**
- state the precision of the thermometer to be used
- use styrofoam cup as the container for the solution mixture

1 mark for describing the followings in the procedure:

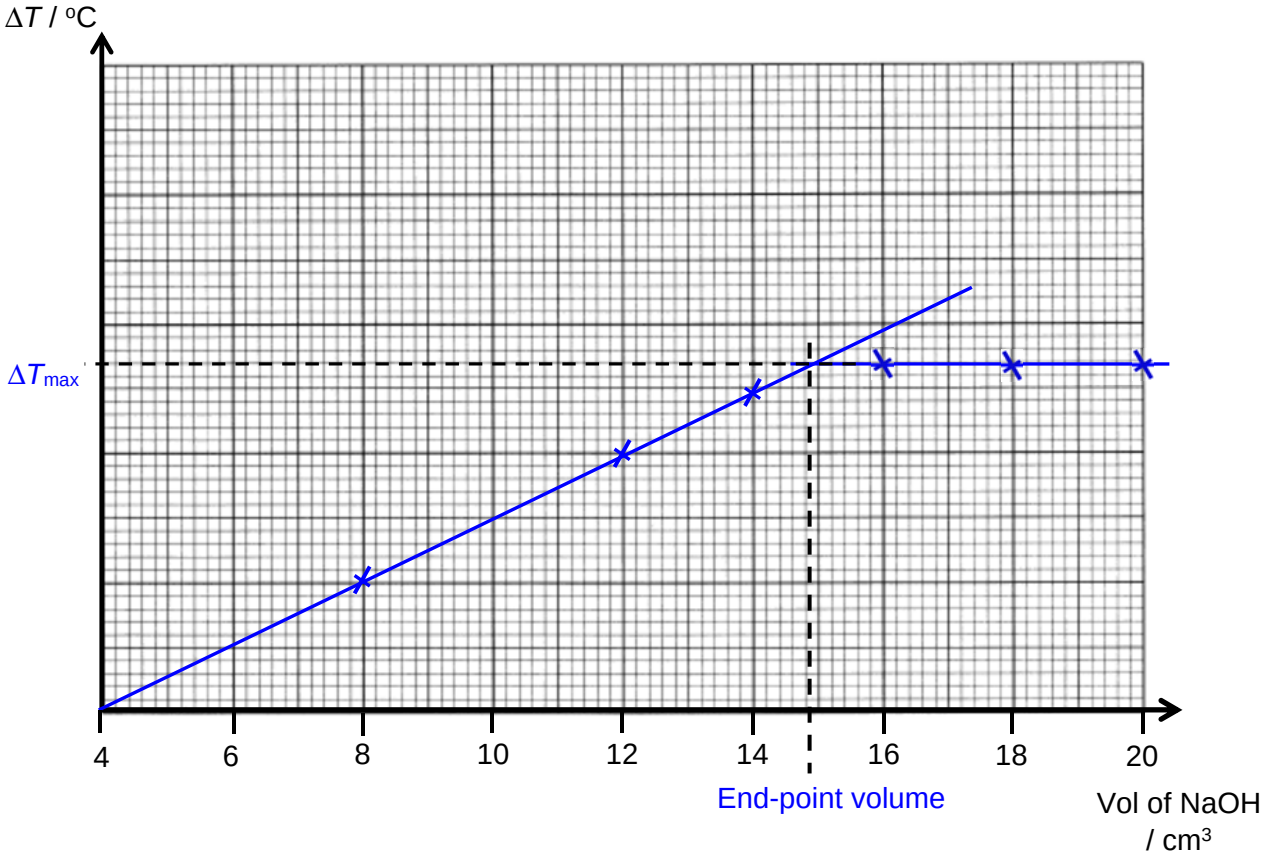
- measure out a fixed volume of **FB 1** and determine its initial temperature
- mix **FB 1**, NaOH and water (either **FB 1** or NaOH should be the last reagent to be added) and record the highest temperature obtained
- repeat the experiment by varying the volumes of NaOH and water to be added and ensuring that the total volume of the reaction mixture remains constant

1 mark for selecting an appropriate fixed volume of **FB 1** and range of volume of water and NaOH taking into account the followings:

- The minimum fixed volume of reaction mixture is 25.00 cm^3 so that the bulb of the thermometer is completely immersed into the solution when measuring the initial temperature
- Using the approximate $\mathbf{V_{max}}$**(FB 2)** calculated to select the appropriate range of the volume of NaOH to be added, i.e. 3 volumes less than 14 cm^3 and 3 volumes greater than 14 cm^3
- Adding appropriate volumes of water to ensure that the total volume of the reaction mixture is fixed and preferably be less than 100 cm^3 taking into account the maximum volume of the polystyrene cup used and to prevent spillage upon mixing

1 mark for writing step **8** in the above procedure

Sketch of graph expected to be obtained:



Marking Scheme

1 mark for

- labelling the axes correctly and indicating the scales on the x axis
- indicating the end-point volume of NaOH

1 mark for drawing a linear, increasing line and a linear, horizontal line. These lines are then extrapolated until they cross. The point of intersection is the equivalence point.

Explanation of the shape of the graph:

Before equivalence point, the limiting reagent is NaOH, hence both the number of moles of water and the total volume of the solution are increasing. According to $\Delta H_n = -\dfrac{m \times c/1000 \times \Delta T}{n_{H_2O}}$, ΔT is directly proportional to the number of moles of water.

Since the number of moles of water increases with increasing volume of NaOH so ΔT increases.

After the equivalence point, the limiting reagent is **FB 3**. The number of moles of water which depend on **FB 3** remain constant as a fixed volume of **FB 3** is used. Hence ΔT remains unchanged.

1 mark for sensible explanation

(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]

[1]: Line rises more steeply and intersects second line at a higher temperature rise
[1]: Maximum is reached at ½V(NaOH) needed to completely neutralised the ethanedioic acid in **FB 1**
[1]: Some of the heat that would have been released is used to ionise the ethanedioic acid and the reacting mole ratio between hydrochloric acid and sodium hydroxide is 1 : 1.

[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.

The temperature, T_1, of the hot water at t = 4 min is 72.0……. °C.

Table of results

time / s	0	1	2	3	5	5.5	6	6.5
temperature / °C	31.5	31.5	31.5	31.5	49.0	49.0	48.5	48.0

time / s	7	7.5	8
temperature / °C	48.0	47.5	47.0

[3]

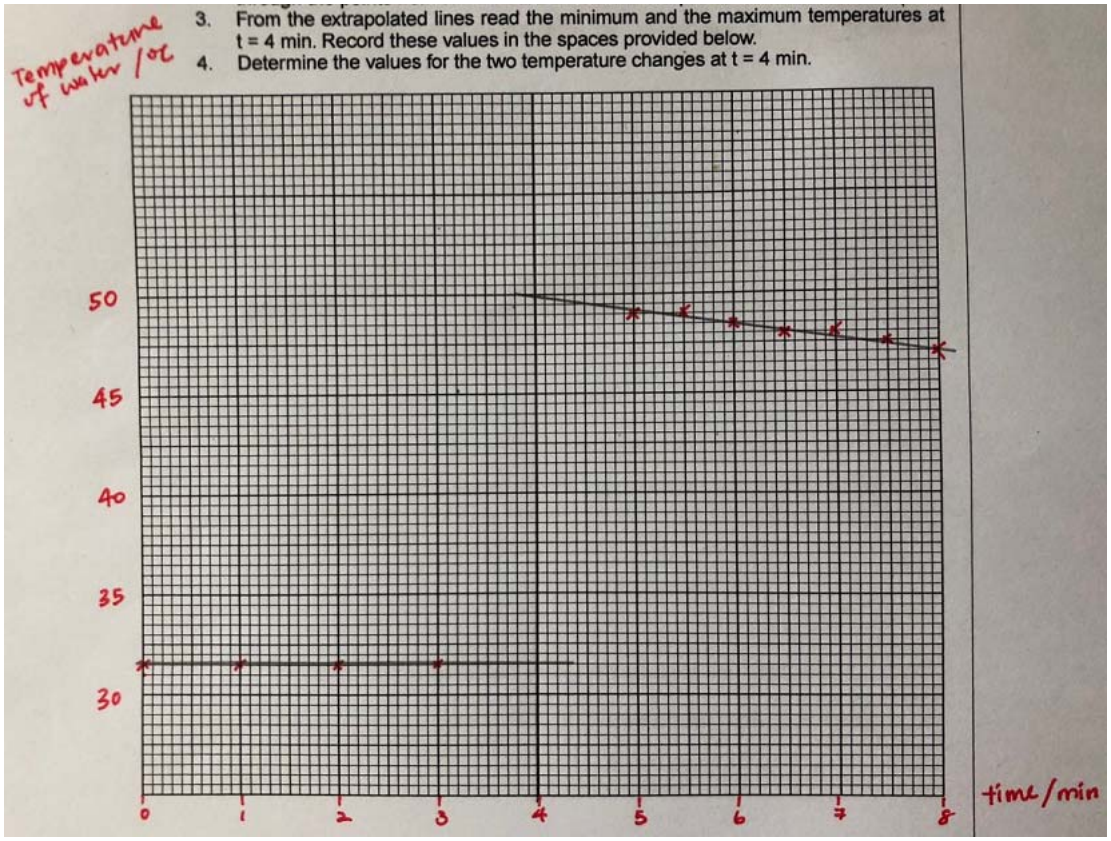
[1]: Follows instructions with regard to times and temperature readings 0-3 minutes at 1 minute intervals; 5-8 minutes at ½ minute intervals, **and T_1** recorded in box. (Ignore if also in table)

[1]: All columns correctly labelled with appropriate unit shown.
Must use solidus, brackets or describe fully in words.
If units not included in column headings every entry must have correct unit shown
Accept min, mins, minutes

[1]: All **thermometer readings** (table and box) recorded to nearest 0.5 °C.
Ignore the precision for the recording of time.

(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, 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.



Minimum temperature, T_2, at t = 4 min is 31.5..... °C.
Maximum temperature, T_3, at t = 4 min is 50.0..... °C.
Temperature rise for 50 cm^3 of cold water in the 100 cm^3 metal calorimeter, ($T_3 - T_2$) is 18.5..... °C.
Temperature fall for 50 cm^3 of hot water from the 100 cm^3 beaker, ($T_1 - T_3$) is 22.0..... °C. [4]

[1]: Temperature of water in the metal calorimeter plotted on y-axis against time on x-axis.
NYJC mark scheme: Clearly labelled axes. Units required. Allow ECF from how headers are labelled in table in **(a)**.
*CIE mark scheme and FYI only: Clearly labelled axes (ignore units) [temp/time are minimum acceptable labels] but accept **T / °C** and **t / min** as labels. The unit is necessary in this case*

[1]: Uniform and sensible scales for candidate's choice of graph.
Plotted points must be in at least 4 large squares on the temperature axis and 5 large squares on the time axis.
Do **not** include any plotted value of T_1.

[1]: There should be a minimum of 5 plotted points between 5 and 8 minutes.
Examiner then checks plotting of points at t_0 min, t_5 min and t_8 min and the plotting of any suspect point.
If any of the t_0 min, t_5 min and t_8 min points is missing check the adjacent point.
Points should be within ½ of a small square of the correct position and in the correct small square

[1]: Acceptable straight lines drawn – an acceptable straight line is one passing through the majority of points or has balanced points on either side of the line
and
correct values of T_2 and T_3 read (to within ½ small square) from the graph.

Extrapolation need not be drawn on the graph

(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.

heat energy gained by cold water = 50 × 4.2 × 18.5 = 3885 ≈ 3890 J

The heat energy gained by the cold water = 3890……. J.

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

heat energy lost by hot water = 50 × 4.2 × 22.0 = 4620 J

The heat energy lost by the hot water = 4620……. 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.

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

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.

approximate heat capacity of the 100 cm^3 metal calorimeter

$$= \frac{4620 - 3885}{18.5}$$

$$= 39.72$$

$$\approx 39.7 \text{ J °C}^{-1}$$

The approximate heat capacity of the 100 cm^3 metal calorimeter = 39.7……. J $°C^{-1}$.

[1]

(i) and **(ii)**

Award one mark if both of the following expressions are correctly evaluated.

heat gained = 210 × candidate value of ($T_3 - T_2$)

heat lost = 210 × candidate value of ($T_1 - T_3$)

Units should be consistent. Ignore any sign given.

(iii) no mark.

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

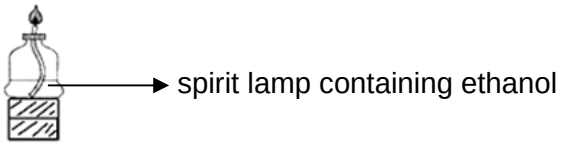
An experiment was carried out as follows to determine the enthalpy change of combustion of ethanol.

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.

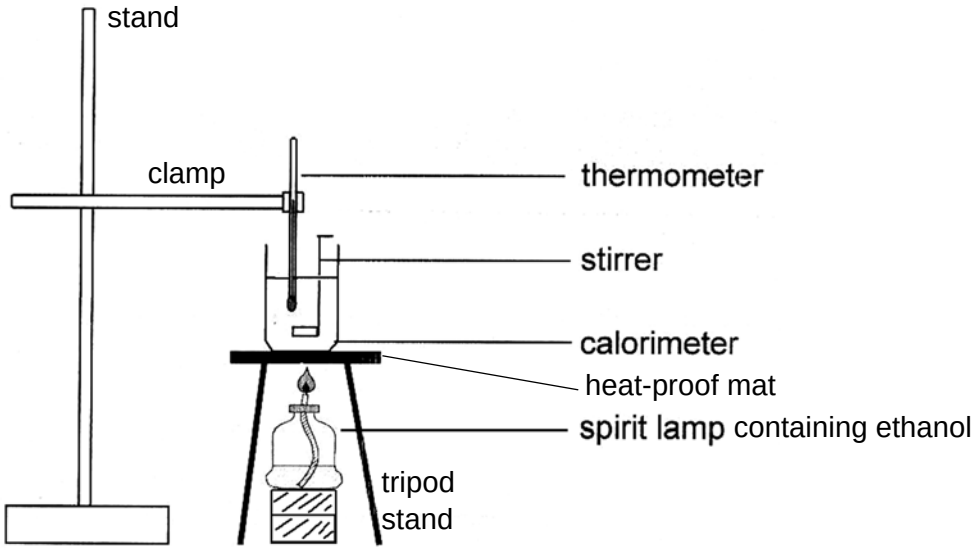
mass of ethanol burned = 0.391 g
mass of water heated = 40.0 g
temperature rise = 19.5 ^oC

(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 $^{o}C^{-1}$.)

ΔH_c(ethanol)

$$= -\frac{(40.0\times 4.2\times 10^{-3}\times 19.5)+(39.72\times 10^{-3}\times 19.5)}{0.391/46.0}$$

$= -477$ kJ mol^{-1}

enthalpy change of combustion of ethanol = –477 kJ mol^{-1}……[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	(0.0005×2)/0.391*100 = 0.256 %
40.0 g of water	0.05 g	0.05/40.0*100 = 0.125 %

[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.

(i)	To 1 cm depth of aqueous **FB 4** in a test-tube, add aqueous ammonia the	White ppt soluble in excess $NH_3(aq)$ [M1]
	add sulfuric acid dropwise until there is no further observations.	White ppt reforms, soluble in excess [M2]
(ii)	To 1 cm depth of aqueous **FB 4** in a test-tube, add silver nitrate,	White ppt formed, soluble in excess $NH_3(aq)$ [M3]
	followed by aqueous ammonia.	

For Examiner's Use			
Obs points	9-11 pts 6 mks 7-8 pts 5 mks 5-6 pts 4 mks 4 pts 3mks 3 pts 2 mks 1-2 pts 1 mk	*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)**.	White solid melts to give colourless liquid. [M4] Colourless, odourless gas relights a glowing splint. Gas is O_2. [M5] White residue obtained on cooling [M6]
(ii)	To the remaining half of **FB 5** is the boiling tube, add 3 cm depth of aqueous sodium hydroxide. Warm gently and retain for the test in **(iii)**.	No NH_3 gas given off. [M7]
(iii)	To the solution from the test in **(ii)**, add a piece of aluminium foil and warm gently.	Pungent colourless gas turns moist red litmus blue. (Gas is NH_3.) [M8]
(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.	Solution turns yellow/brown/black solid formed [M9]
(vi)	To the second part of the solution from **(iv)**, add aqueous potassium manganate (VII) followed by dilute sulfuric acid.	Purple MnO_4^- solution decolourised [M10]
(vii)	To the third part of the solution **(iv)**, add aqueous sodium hydroxide and a piece of aluminium foil. Warm gently.	Pungent colourless gas turns moist red litmus blue. (Gas is NH_3.) [M11]

[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]

Zn^{2+} & Cl^- [1]

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

……

……

……

……

……

……

…… [3]

When aq NH_3 is added, a white ppt of $Zn(OH)_2$ is formed.

- $Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2$ ……. (1)
- When excess aqueous NH_3 is added, ligand exchange takes place. NH_3 ligand replaces water ligand from $[Zn(H_2O)_6]^{2+}$ to form $[Zn(NH_3)_4]^{2+}$ which is a colourless solution.
- $[Zn(H_2O)_6]^{2+} + 4NH_3 \rightarrow [Zn(NH_3)_4]^{2+} + 6H_2O$ ……. (2)

Accept $[Zn(NH_3)_6]^{2+}$ and $[Zn(NH_3)_4(H_2O]_2]^{2+}$ and pls mark equation (2) accordingly.

- This causes $[Zn(H_2O)_6]^{2+}$ to drop, hence equilibrium (1) shifts to the left, and the precipitate, $Zn(OH)_2$ dissolves.

  (The drop in $[Zn(H_2O)_6]^{2+}$ also causes the ionic product of $Zn(OH)_2$ to decrease to the extent that IP of $Zn(OH)_2$ < Ksp of $Zn(OH)_2$. Hence the precipitate, $Zn(OH)_2$ dissolves.)

- When $H_2SO_4(aq)$ was added, $[NH_3]$ decreases, hence equilibrium (1) shifts to the left, and the precipitate, $Zn(OH)_2$ reforms.
- precipitate dissolves due to <u>further</u> removal of OH^- by the <u>excess</u> $H_2SO_4(aq)$, causing the POE of reaction (2) to shift to the left.

3 marks for 6 bullets correct
2 marks for 3, 4-5 bullets correct
1 mark for 1-2 bullets correct

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

…… [1]

NO_3^- [1]

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

…… [1]

NO_2^- [1]

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

an oxidising agent [1]

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

a reducing agent [1]

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

……

……

…… [1]

- To remove any NH_4^+ ions that is present as NH_3, as the presence of NH_4^+ will interfere with the results of **(b)(iii)**.

OR

- to confirm that there is no NH_4^+ ions present in the sample, so that there will be no interference of the results of **(b)(iii)**.

1 mark

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

Experiment	Observation
To solid **FB 5** in a boiling tube. Heat heat strongly for 4 min. Let the residue cool. Add dilute sulfuric acid to it.	Brown pungent gas formed (Gas is NO_2.)

[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*È(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, BrÈ(aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, IÈ(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq))
nitrate, NO_3È(aq)	NH_3 liberated on heating with OHÈ(aq) and A*l* foil
nitrite, NO_2È(aq)	NH_3 liberated on heating with OHÈ(aq) and A*l* foil; NO liberated by dilute acids (colourless NO → (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