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Paper 4 Practical Paper

Candidates answer on question paper.

READ THESE INSTRUCTIONS FIRST

Write your name and class in the spaces at the top of this page. Give details of the practical shift and laboratory where appropriate, in the boxes provided.

SUGGESTED ANSWERS

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 23 and 24.

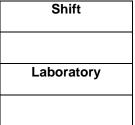
At the end of the examination, fasten all your work securely together. The number of marks is given in brackets [] at the end of each question or part question.

YISHUN INNOVA JUNIOR COLLEGE JC 2 PRELIMINARY EXAMINATION

Shift
 Laboratory

20 August 2024 2 hours 30 minutes

For Examiner's use		
1		
2		
3		
4		
Total		





CANDIDATE

NAME

CLASS

9729/04

2

Answer all the questions in the spaces provided.

1 Determination of the kinetics of a redox reaction

Equation 1 represents the reaction between iron(III) ions and iodide ions.

equation 1 $2Fe^{3+}(aq) + 2I^{-}(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$

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

If a small amount of sodium thiosulfate, $Na_2S_2O_3$, is also present in the reaction mixture, the formation of the blue colour is delayed. The $Na_2S_2O_3$ reacts with I_2 as shown in equation 2.

equation 2 $I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^{-}(aq)$

FA 1 is iron(III) chloride, FeC l_3 . FA 2 is potassium iodide, KI. FA 3 is 0.0060 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃. Solution S is starch indicator.

You will perform a series of five experiments. Then, you will graphically analyse your results to determine the order with respect to the concentration of Fe³⁺ ions, [Fe³⁺].

You will add a fixed amount of sodium thiosulfate, **FA 3**, to each of your experiments. In each experiment, you will need to ensure that the same total volume of reaction mixture is used by adding deionised water as required. The time taken for the blue colour to form allows the reaction rate to be determined.

- (a) Prepare a table in the space provided on page 4 in which to record, to an appropriate level of precision:
 - all volumes, except the volume of sodium thiosulfate, potassium iodide and starch,
 - all values of t.

Experiment 1

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

- 1. Fill a burette with **FA 1**.
- 2. Transfer 20.00 cm³ of FA 1 into a 250 cm³ beaker labelled "reaction mixture".
- 3. Use appropriate measuring cylinder, transfer to a clean 100 cm³ beaker.
 - 10.0 cm³ of **FA 2**,
 - 10.0 cm³ of **solution S**,
 - 20.0 cm³ of **FA 3**.
- 4. Pour this mixture rapidly into the beaker containing **FA 1** and stir the contents using a glass rod. Start the stopwatch during this addition.
- 5. The mixture turns brown and then yellow before turning a blue colour. Stop the stopwatch when the solution **first** turns blue.
- 6. Record the time taken, *t*, to the nearest second in your table.
- 7. Carefully wash out the beaker. Stand it upside down on a paper towel to drain.

Experiment 2

- 1. Fill another burette with deionised water.
- 2. Transfer 6.00 cm³ of **FA 1** into another 250 cm³ beaker labelled "reaction mixture".
- 3. Transfer 14.00 cm³ of deionised water into the beaker containing **FA 1**.
- 4. Repeat points 3 7 from experiment 1.

Experiment 3 to 5

Carry out **three** further experiments to investigate the effect of changing the concentration of $Fe^{3+}(aq)$ by changing the volume of aqueous **FA 1** used.

You should **not** use a volume of **FA 1** that is less than 6.00 cm³.

In each case, you will need to ensure that the same total volume of reaction mixture is used by adding deionised water as required.

You should alternate the use of the two 250 cm³ beakers labelled "reaction mixture".

Record all required volumes and time taken in your table.

Results

experiment	V _{FA1} / cm ³	V _{deionised water} / cm ³	time/ s
1	20.00	0.00	15
2	6.00	14.00	24
3	10.00	10.00	21
4	14.00	6.00	17
5	17.00	3.00	16

- (b) The rate of reaction can be found by calculating the change in concentration of Fe³⁺(aq) that occurred when enough iodine was produced to change the colour of the indicator to blue.
 - (i) Calculate the amount, in moles, of thiosulfate ions, $S_2O_3^{2-}$, used in each experiment.

Amount of $S_2O_3^{2-} = \frac{20}{1000} \times 0.00600 = 1.20 \times 10^{-4} \text{ mol}$

(ii) Calculate the amount of iron(III) ions, Fe³⁺, that were used to produce the amount of iodine that react with the amount of S₂O₃²⁻ in (b)(i).

Mole ratio of $S_2O_3{}^{2-}$: I_2 : Fe^{3+} is 2 : 1 : 2 Amount of Fe^{3+} = 1.20 \times 10^{-4} mol

(iii) Calculate the change in the concentration of iron(III) ions up to the time of appearance of the blue colour.

Change in $[Fe^{2+}] = (1.20 \times 10^{-4}) \div (60 \div 1000) = 2.00 \times 10^{-4} \text{ mol dm}^{-3}$

(iv) The rate of this reaction relative to $[Fe^{3+}]$ can be determine using the following expression.

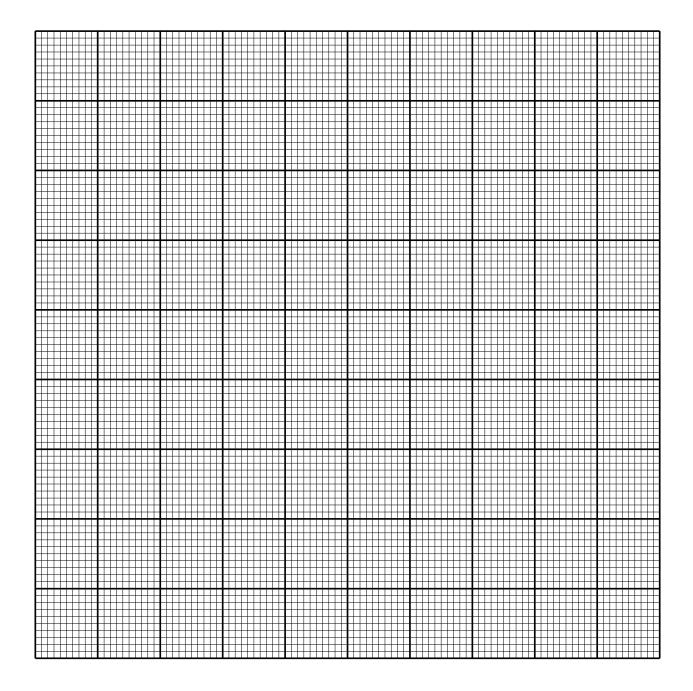
rate =
$$\frac{\text{change in concentration of Fe}^{3+}(aq)}{\text{reaction time}} \times 10^{6}$$

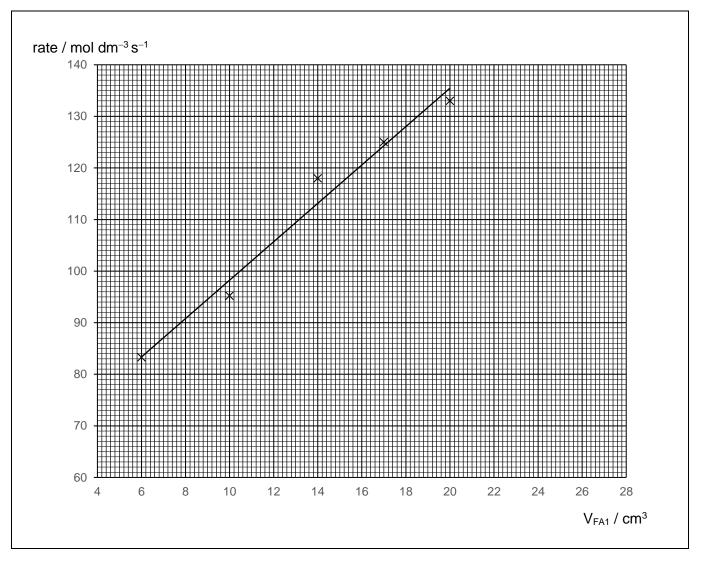
Complete the table to show the volume of **FA 1**, the reaction time and the rate in Experiments 1 to 5. You should include units.

If you were unable to calculate a value for the change in concentration of $Fe^{3+}(aq)$ in **(b)(iii)**, you should assume it is 2.50 × 10⁻³ mol dm⁻³. Note that this is not the correct value.

experiment	V _{FA1} / cm ³	reaction time / s	rate / mol dm ⁻³ s ⁻¹
1	20.00	15	133
2	6.00	24	83.3
3	10.00	21	95.2
4	14.00	17	118
5	17.00	16	125

(c) (i) Plot a graph of rate on the *y*-axis against volume of **FA 1** on the *x*-axis. Draw the best-fit straight line taking into account all of your plotted points.





(ii) Deduce the order of reaction with respect to [Fe³⁺]. Use evidence from your graph in (c)(i) to support your answer.

Reaction is first order with respect to $[Fe^{3+}]$. The straight-line graph shows that rate is proportional to $[Fe^{3+}]$ (or volume of Fe^{3+}). (d) It was found by carrying out experiments similar to those used in (a), that increasing the concentration of I⁻ increased the rate of the reaction.

A student suggested a modification to the method by using the same volumes of all reagents but with the concentrations of **FA 1** and **FA 2** being double their original values.

State what the effect would be on the reaction time in Experiment 1 and explain how this change would affect any possible errors in the measurements.

Reaction time is less/shorter (not faster).

The measurement will be less accurate since there is a larger percentage error in time.

(e) Another student thought that the experiment could be made more accurate by repeating Experiment 1 with the same volumes of all reagents but using 0.060 mol dm⁻³ sodium thiosulfate instead of FA 3. He found that the reaction mixture never turned blue.

Explain why.

 $S_2O_3^{2-}$ is in excess. All the iodine reacts with $S_2O_3^{2-}$ so no iodine is left to turn starch solution blue.

2 Investigation of an inorganic and organic compound

FA 4 contains one cation and one anion.

FA 5 is a solution of an aromatic organic compound, $C_7O_3H_6$, which contains two different functional groups.

You will perform tests to identify:

- the ions present in **FA 4**,
- the functional groups in **FA 5** and hence deduce its possible structure.

The volumes given below are approximate and should be estimated rather than measured.

Test and identify any gases evolved.

If there is no observable change, write **no observable change**.

(a) (i) Carry out the following tests. Carefully record your observations in **Table 2.1**.

	test	observations
1	Test the FA 4 solution using Universal Indicator paper.	Orange colour on UI paper corresponds to pH 3.
2	Add 1 cm depth of FA 4 to a test-tube. Add aqueous sodium hydroxide, slowly with shaking, until no further change is seen.	white ppt soluble in excess
3	Add 1 cm depth of FA 4 to a test-tube. Add aqueous ammonia, slowly with shaking, until no further change is seen.	white ppt insoluble in excess

Table 2.1

(ii) Identify the cation in **FA 4** and state the evidence to support your deduction.

cation	evidence	
A <i>l</i> ³⁺	It forms a white precipitate with NaOH(aq) which is soluble in excess and forms a white precipitate with $NH_3(aq)$ which is insoluble in excess.	
		[1]

(iii) Write ionic equations to account for your observations in test 2.

 $Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Al (OH)_{3}(s)$

 $Al (OH)_3(s) + OH^- (aq) \rightarrow [Al(OH)_4]^-(aq)$

(iv) Devise and perform a series of simple tests to identify the anion in **FA 4**. Your test should be based on the Qualitative Analysis Notes on pages 23 – 24 and should use only the bench reagents provided. Record your tests and observations in the space below.

Any test requiring heating MUST be performed in a boiling tube.

FA 4 does not contain carbonate, nitrite or any sulfur ions.

test	observations
To 1 cm depth of FA 4 , add a few drops of AgNO ₃ .	No observable change
To 1 cm depth of FA 4 in a boiling tube, add 1 cm depth	(White ppt formed, soluble in excess NaOH to form a colourless solution.)
of aqueous NaOH and a piece of A <i>l</i> foil. Heat cautiously.	Upon heating, gas evolved turned damp red litmus paper blue.

(v) Use your observations in (a)(iv) to deduce the identity of the anion in FA 4.

NO ₃ ⁻	
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(b) (i) Carry out the following tests. Carefully record your observations in Table 2.2.

Table 2.2

To a 2 cm depth of FA 5 in a test tube, add a small spatula of sodium carbonate. To a 1 cm depth of aqueous iron(III) chloride in a test-tube, add 10 drops of FA 5 .	Effervescence observed. Gas gives a while precipitate with limewater. Violet solution observed.
chloride in a test-tube, add 10 drops of	Violet solution observed.
Do not perform the following test. Observations have been recorded for you.	
To a 1 cm depth of FA 5 in a test tube, add an equal depth of aqueous promine.	Orange solution turns colourless. White precipitate formed.
C fe	Observations have been recorded or you. To a 1 cm depth of FA 5 in a test tube, dd an equal depth of aqueous

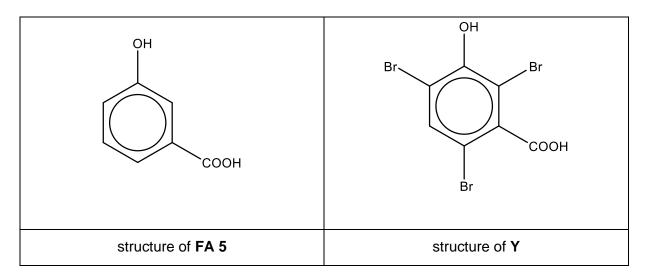
(ii) Consider your observations in Table 2.2.

Name the functional group that can be identified using aqueous iron(III) chloride and aqueous bromine.

phenol

(iii) In test 3, the white precipitate, **Y**, with $M_r = 374.7$ is formed. [A_r of C = 12.0, H = 1.0, O = 16.0, Br = 79.9]

Consider your observations and deductions from **Table 2.2**, suggest the structure of **FA 5** and **Y**.



3 Determination of a value for the relative atomic mass, Ar, of magnesium

FA 6 is 2.00 mol dm⁻³ hydrochloric acid, HC*l*.
FA 7 is 0.120 mol dm⁻³ sodium hydroxide, NaOH Magnesium ribbon
Solution I is bromophenol blue indicator

You are to react magnesium with **FA 6**, dilute the reaction mixture and titrate this diluted sample with **FA 7**. The data from the titration will be used to calculate the relative atomic mass, A_r , of magnesium.

(a) (i) Procedure

- 1. Pipette 25.0 cm³ of **FA 6** into a 250 cm³ beaker.
- 2. Weigh the magnesium ribbon and record its mass.

mass of magnesium = 0.192 g

- 3. Coil the magnesium ribbon loosely and add it to the FA 6 in the beaker.
- 4. Stir the reaction mixture occasionally and wait until the reaction is completed.
- 5. Transfer all the solution from the beaker into a 250 cm³ volumetric flask.
- 6. Make the solution up to the mark with deionised water and shake well to mix.
- 7. Label this solution **FA 8**.
- 8. Fill a clean burette with FA 7.
- 9. Rinse the pipette thoroughly.
- 10. Pipette 25.0 cm³ **FA 8** into a 250 cm³ conical flask.
- 11. Add a few drops of **solution I** to the conical flask.
- 12. Run **FA 7** from the burette into the conical flask. The end-point is reached when the solution just becomes green.
- 13. Record your titration results to an appropriate level of precision in the space on page 15.
- 14. Repeat steps 10 to 13 until consistent results are obtained.

14

Titration results

titration number	1	2
final burette reading / cm ³	29.30	29.30
initial burette reading / cm ³	0.00	0.00
volume of FA 7 used / cm ³	29.30	29.30

(ii) From your titrations, obtain a suitable volume of **FA 7** to be used in your calculations. Show clearly how you obtained this volume.

Volume of FA 7 used = (29.30 + 29.30) ÷ 2 = 29.30 cm³

(b) (i) Calculate the amount, in moles, of hydrochloric acid added to the magnesium ribbon during the preparation of **FA 8**.

Amount of HCl added = $\frac{25}{1000} \times 2.00 = 0.0500$ mol

(ii) Use your titration results to calculate the amount of hydrochloric acid in the volumetric flask after the reaction with magnesium during the preparation of FA 8. Hence calculate the amount of hydrochloric acid that reacted with the magnesium.

Amount of NaOH = $\frac{29.30}{1000} \times 0.120 = 0.003516$ mol Amount of HCl in 25.0 cm³ **FA8** = 0.003516 mol Amount of HCl in 250 cm³ **FA8** = $\frac{250}{25.0} \times 0.003516 = 0.03516 = 0.0352$ mol Amount of HCl reacted = 0.0500 - 0.03516 = 0.01484 = 0.0148 mol 15

(iii) The reaction between magnesium and hydrochloric acid is shown.

 $Mg(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2(g)$

Calculate the relative atomic mass, Ar, of magnesium.

Amount of Mg = $0.5 \times 0.01484 = 0.00742$ mol $A_r = 0.192 \div 0.00742 = 25.9$

(c) (i) A student follows the procedure described and calculated the relative atomic mass of magnesium to be 24.1.
The theoretical value of the relative stemic mass of magnesium is 24.2.

The theoretical value of the relative atomic mass of magnesium is 24.3.

Calculate the difference between the theoretical value and the student's calculated value as a percentage. This is the percentage error.

percentage error = $\frac{24.3-24.1}{24.3} \times 100\% = 0.823\%$

(ii) The student calculates that the maximum total percentage error of the whole procedure is 1.09%.

Did the student perform the experiment well? Explain your answer.

Performed well as the percentage error of the A_r obtained is less than the total percentage error of the whole procedure.

4 Planning

Acetic acid, CH₃CO₂H, can be found in vinegar and provides its signature tangy flavour to enhance culinary dishes.

Acetic acid reacts with sodium hydroxide, NaOH, according to equation 1.

equation 1 $CH_3CO_2H(aq) + NaOH(aq) \rightarrow CH_3COONa(aq) + H_2O(I) \Delta H_n$

When CH₃CO₂H is mixed with NaOH, the neutralisation reaction releases heat causing a rise in the temperature of the solution.

A series of experiments can be carried out using different volumes of the two solutions, CH_3CO_2H and NaOH. In each experiment, the total volume of the two solutions is to be kept constant at 50 cm³ and the change in temperature, ΔT , is to be determined. Since the total volume of mixture remains the same, the temperature rise, ΔT , is a direct measure of the heat liberated by the reaction. The maximum amount of heat is evolved when all the acid present is exactly neutralised by all the alkali present.

Plotting a graph of ΔT against the volume of CH₃CO₂H used will give 2 straight lines of best–fit.

Extrapolation of the two straight lines will produce a point of intersection from which the concentration of CH₃CO₂H and the enthalpy change of neutralisation, ΔH_n , between CH₃CO₂H and NaOH can be determined.

(a) In an experiment, 1.00 mol dm⁻³ of NaOH was reacted with acetic acid and the total volume of the two solutions used was 50 cm³.

Assuming that the concentration of acetic acid is approximately 0.880 mol dm⁻³, calculate the volume of acetic acid to be used so that stoichiometric amounts of acetic acid and NaOH react.

Let volume of acetic acid used be $V \text{ cm}^3$. $\frac{V}{1000} \times 0.880 = \frac{(50 - V)}{1000} \times 1.00$ 0.880V = 50 - V $V = 26.6 \text{ cm}^3$ (b) Using the information given, you are required to write a plan to determine the concentration of acetic acid in vinegar, and the value of ΔH_n using a graphical method.

You may assume that the original solutions are at room temperature.

You may also assume that you are provided with:

- 250 cm³ of approximately 0.880 mol dm⁻³ acetic acid
- 250 cm³ of 1.00 mol dm⁻³ NaOH(aq),
- the equipment normally found in a school or college laboratory.

Your plan should include:

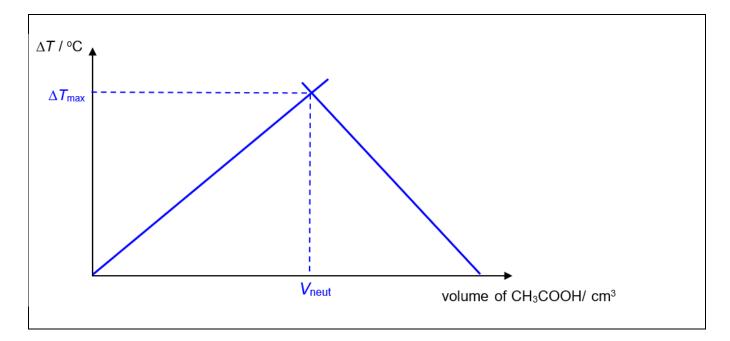
- brief, but specific, details of the apparatus you would use, bearing in mind the levels of precision they offer,
- the quantities you would use,
- the procedure you would follow,
- the measurements you would make to allow a suitable ΔT against the volume of CH₃CO₂H graph to be drawn.
- 1. Place a clean and dry polystyrene cup inside a second polystyrene cup which is placed in a 250 cm³ beaker (to prevent the cups from tipping over).
- 2. Using a 50.00 cm³ burette, transfer 10.00 cm³ of CH₃CO₂H(aq) to a 100 cm³ beaker. Measure the initial temperature, T_1 of MOH. Wash and dry the thermometer
- 3. Using another burette, transfer 40.00 cm³ of NaOH(aq) into the polystyrene cup and put a lid on.
- 4. Insert a 0.2 °C thermometer through the lid and ensure that the bulb of the thermometer is fully immersed in NaOH(aq). Measure the initial temperature, T_2 , of NaOH(aq).
- 5. Open the lid of the cup and transfer the 10.00 cm³ of CH₃CO₂H(aq) into the polystyrene cup containing NaOH(aq). Close the lid quickly.
- 6. Stir the mixture continuously using the thermometer until the highest temperature is reached. Record this temperature, T_3 .
- 7. Discard the contents of the polystyrene cup.
- 8. Wash and dry the polystyrene cup and thermometer.
- 9. Repeat step 1 to 8 using the following volumes.

Volume of CH ₃ CO ₂ H / cm ³	Volume of NaOH / cm ³
15.0	35.0
20.0	30.0
25.0	25.0
30.0	20.0
35.0	15.0
40.0	10.0

10. Record all measurements of volume, initial temperatures, calculated weighted average of the initial temperatures and temperature change, ΔT , in an appropriate format.

- (c) Sketch on Fig. 4.1, the graph you would expect to obtain from your results. Indicate clearly on your sketch how you would determine:
 - V_{neut} , the volume of CH₃CO₂H used
 - ΔT_{max} , the maximum temperature change

when stoichiometric amount of CH₃CO₂H and NaOH react.



- (d) Outline how you would use your answers from (c) to determine:
 - (i) the concentration of acetic acid in vinegar, in mol dm⁻³. Express your results in terms of V_{neut} .

Volume of NaOH = $(50 - V_{neut})$ Mole ratio of CH₃CO₂H : NaOH = 1 : 1 Amount of CH₃CO₂H = Amount of NaOH $(50 - V_{neut}) \times 1.00 = V_{neut} \times [CH_3CO_2H]$ [CH₃CO₂H] = $\frac{(50 - V_{neut})}{V_{neut}}$ (ii) the enthalpy change of neutralisation, ΔH_n . Express your results in terms of ΔT_{max} and V_{neut} .

You may assume the following:

- specific heat capacity of all solutions is 4.18 J g⁻¹ K⁻¹,
- density of all solutions is 1.00 g cm⁻³.

Heat transferred = $(50 \times 4.18 \times \Delta T_{max}) / 1000 \text{ kJ}$ Amount of H₂O formed = amount of NaOH = $1.00 \times \frac{(50 - V_{neut})}{V_{neut}}$ $\Delta H_{neut} = -[(50 \times 4.18 \times \Delta T_{max}) / 1000] \div [\frac{(50 - V_{neut})}{V_{neut}}]$ $= -(209 \times \Delta T_{max}) / (50 - V_{neut}) \text{ kJ mol}^{-1}$

(e) A similar exothermic reaction takes place between HCl(aq) and NaOH(aq).

Explain how the ΔT_{max} would differ from the value obtained in (c) when the same experiment described in (b) is repeated using HC*l*(aq) of the same concentration as acetic acid.

 ΔT_{max} will be higher as acetic acid is a weak acid OR HCl is a strong acid.

Energy will be absorbed to allow complete dissociation of acetic acid molecules OR HC*l* dissociates completely in solution.

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	reaction with		
cation	NaOH(aq)	NH₃(aq)	
aluminium, A <i>l</i> ³+(aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH₄⁺(aq)	ammonia produced on heating	-	
barium, Ba²⁺(aq)	no ppt. (if reagents are pure)	no ppt.	
calcium, Ca²+(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), Cu²+(aq),	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution	
iron(II), Fe²+(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), Mn²+(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, Zn²⁺(aq)	white ppt. soluble in excess	white ppt. soluble in excess	

(b) Reactions of anions

ions	reaction		
carbonate, CO3 ²⁻	CO ₂ liberated by dilute acids		
chloride, C <i>l</i> ⁻(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, NO₃⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil		
nitrite, NO₂⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)		
sulfate, SO₄²⁻(aq)	gives white ppt. with Ba ²⁺ (aq) (insoluble in excess dilute strong acids)		
sulfite, SO₃²⁻(aq)	SO ₂ liberated with dilute acids; gives white ppt. with Ba ²⁺ (aq) (soluble in dilute strong acids)		

(c) Test for gases

ions	reaction	
ammonia, NH₃	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, I2	black solid / purple gas	brown	purple