

HWA CHONG INSTITUTION C2 Preliminary Examination Higher 2

NAME

CT GROUP

21S

CHEMISTRY

Paper 4 Practical

9729/04 25 August 2022 2 hours 30 minutes

Candidates answer on the Question Paper

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 16 and 17.

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		
2		
3		
4		
Total		

1 Determination of the basicity of an acid and the strength of a base

FA 1 is 1.50 mol dm⁻³ sodium hydroxide, NaOH.

FA 2 is 0.75 mol dm⁻³ acid **A**, which is either monobasic (monoprotic) or dibasic (diprotic). **FA 3** is 1.50 mol dm⁻³ base **B**, which is monoacidic (monoprotic) and is either a strong base or a weak base.

You will perform experiments to determine whether acid **A** is monobasic or dibasic. You will then calculate the enthalpy change of neutralisation, ΔH_{neut} , between NaOH and acid **A**.

You will also design one more experiment to determine whether base ${f B}$ is a strong base or a weak base.

(a) (i) Experiment 1

- 1. Place one polystyrene cup inside a second polystyrene cup. Place these in a glass beaker to prevent them from tipping over.
- Use a measuring cylinder to transfer 40.0 cm³ of FA 1 into the first polystyrene cup. Stir the FA 1 in the polystyrene cup with the thermometer with 0.2 °C divisions. Read and record the initial temperature of FA 1 in Table 1.1.
- 3. Use another measuring cylinder to measure 20.0 cm³ of **FA 2**. Add **FA 2** to **FA 1** in the polystyrene cup. Stir the mixture using the same thermometer and record the maximum temperature reached.
- 4. Wash and dry the thermometer and the polystyrene cup.

Experiment 2

Repeat experiment 1 using 20.0 cm³ of **FA 1** and 40.0 cm³ of **FA 2**.

Use your results to determine the maximum change in temperature, ΔT , for each experiment and complete Table 1.1. You may assume that the initial temperature of **FA 2** is the same as that of **FA 1**.

experiment	Volume of FA 1 /cm ³	Volume of FA 2 /cm ³	Initial temperature of FA 1 /°C	Maximum temperature /°C	Maximum change in temperature, $\Delta T / ^{\circ}C$
1	40.0	20.0	29.8	32.9	3.1
2	20.0	40.0	29.8	36.0	6.2

Table 1.1

[2]

(ii) Use your ΔT results of experiments 1 and 2 to deduce whether acid **A** is monobasic or dibasic. Explain your deduction.

.....[2]

(iii) Hence, calculate the enthalpy change of neutralisation, ΔH_{neut} , between NaOH and acid A.

Assume that the specific heat capacity of the reaction mixture is 4.18 J g⁻¹ K⁻¹, and that the density of the reaction mixture is 1.00 g cm^{-3} .

 $\Delta H_{\text{neut}} = \dots \dots [2]$

(iv) A student repeated experiment 2 but using solutions of double the concentrations (3.00 mol dm⁻³ NaOH and 1.50 mol dm⁻³ acid **A**).

Suggest the effect this would have on the value of ΔT for experiment 2. Hence, deduce and explain the effect this would have on the value for ΔH_{neut} .

.....[2]



(b) (i) Experiment 3

FA 3 contains a monoacidic base, base B, that has the same concentration as the NaOH in FA 1.

Design one more experiment, the results of which, when compared against **your** experiment 2, will allow you to deduce whether base **B** is a strong or weak base.

Fill Table 1.2 with your chosen volumes of **FA 3** and **FA 2**. Carry out this experiment. Complete Table 1.2.

Table	1.2
-------	-----

experiment	Volume of FA 3 /cm ³	Volume of FA 2 /cm ³	Initial temperature of FA 3 /°C	Maximum temperature /°C	Maximum change in temperature, $\Delta T/^{\circ}C$	
3	20.0	40.0	29.0	34.0	5.0	
						[1

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(ii) Use your ΔT results of experiment 3 and **experiment 2** to deduce whether base **B** is a strong base or a weak base. Explain your deduction.



[Total: 10]

2 Investigation of the kinetics of the catalysed decomposition of hydrogen peroxide

Hydrogen peroxide decomposes very slowly to form water and oxygen as shown in equation 1.

equation 1 $2H_2O_2 \rightarrow 2H_2O + O_2$

Many transition element ions are able to catalyse the decomposition of hydrogen peroxide. Iron(III) nitrate, $Fe(NO_3)_3$, is an effective catalyst for this reaction.

FA 4 is iron(III) nitrate, Fe(NO₃)₃. FA 5 is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄. FA 6 is aqueous hydrogen peroxide, H₂O₂. FA 7 is 0.2 mol dm⁻³ sulfuric acid, H₂SO₄. (FA 7 is also used in **Question 3**.)

You will add a measured volume of **FA 4** to a measured volume of **FA 6** and, at timed intervals, you will transfer aliquots (portions) of the reaction mixture.

It is necessary that you titrate each aliquot against FA 5 before transferring the next aliquot.

Acidified KMnO₄ and H_2O_2 react as shown in equation **2**.

equation 2 $2MnO_4^{-}(aq) + 5H_2O_2(aq) + 6H^{+}(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 5O_2(g)$

(a) (i) Preparation and titration of the reaction mixture

Notes: You will perform each titration **once** only. Great care must be taken that you do not overshoot the end-point.

Once you have started the stopwatch, it must continue running for the duration of the experiment. You must **not** stop it until you have finished this experiment.

You should transfer your first aliquot within the first three minutes of starting the reaction.

The titre volume for your first titration is expected to be greater than 30 cm³.

You should **not** exceed a maximum reaction time of 25 minutes for this experiment.

In an appropriate format in the space provided on the next page, prepare a table in which to record for each aliquot

- the time of transfer, *t*, in minutes and seconds,
- the decimal time, t_d , in minutes, to 0.1 min, for example, if t = 4 min 33 sthen $t_d = 4 \text{ min } + 33/60 \text{ min} = 4.6 \text{ min}$,
- the burette readings and the volume of **FA 5** added.

- 1. Fill a burette with **FA 5**.
- 2. Using a measuring cylinder, add 100.0 cm³ of **FA 6** to the conical flask labelled **reaction mixture**.

Do **not** use the H_2O_2 bench reagent for this step.

- 3. Using a measuring cylinder, add 2.0 cm³ of **FA 4** to the same conical flask. Start the stopwatch and swirl the mixture thoroughly to mix its contents.
- 4. Using a measuring cylinder, add 50.0 cm³ of **FA 7** to a second conical flask.
- 5. Before 3 minutes, withdraw a 10.0 cm³ aliquot (portion) of the reaction mixture to a 10 cm³ measuring cylinder, using a dropping pipette.
- 6. **Immediately** transfer this aliquot into the second conical flask from step 4 and vigorously swirl the mixture. Read and record the time of transfer in minutes and seconds, to the nearest second, when the aliquot is added.
- Immediately titrate the H₂O₂ in the second conical flask with FA 5. The end-point is reached when a permanent pale pink colour is obtained. Record your titration results.
- 8. Wash out the second conical flask with water.
- 9. Repeat steps **4** to **8** until a total of **five** aliquots have been titrated and their results recorded.

You should **not** exceed a maximum reaction time of 25 minutes for this experiment.

tim	ne	t./min	Final Burette	Initial Burette	Vol of FA 5 / cm ³
min	S	ld / IIIII	reading / cm ³	reading / cm ³	
3	1	3.0	35.10	0.10	35.00
7	0	7.0	24.80	0.00	24.80
12	30	12.5	15.40	0.40	15.00
18	7	18.1	25.20	15.60	9.60
23	0	23.0	31.60	25.20	6.40

Results

(a) (ii) Plot a graph of the volume of **FA 5** added, on the *y*-axis, against decimal time, t_d , on the *x*-axis on the grid in Fig. 2.1.

Draw the most appropriate best-fit curve taking into account all of your plotted points.

Extrapolate (extend) this curve to the *y*-axis and read the value of V₀, the volume of **FA 5** required at $t_d = 0.0$ min.



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[Turn over

- (b) The initial rate of change of the concentration of hydrogen peroxide, $[H_2O_2]$, can be determined from the gradient of the tangent to the graph in Fig. 2.1 at time $t_d = 0.0$ min.
 - (i) Draw a tangent to your graph in Fig. 2.1 at time $t_d = 0.0$ min. Determine the gradient of this line, showing clearly how you did this.

gradient = $cm^3 min^{-1}$ [2]

(ii) Use your gradient to determine the rate of change of the amount of MnO_4^- ions required in mol min⁻¹.

rate of change of the amount of MnO_4^- ions required = mol min⁻¹ [1]

(iii) Determine the amount of H_2O_2 decomposed per minute and hence deduce the rate of change of $[H_2O_2]$ at $t_d = 0.0$ min, in mol dm⁻³ min⁻¹.

amount of H_2O_2 decomposed = mol min⁻¹

rate of change of $[H_2O_2]$ at $t_d = 0.0 \text{ min} = \dots \text{ mol } dm^{-3} \text{ min}^{-1}$ [3]

(iv) It has been claimed that the decomposition of hydrogen peroxide is first order with respect to $[H_2O_2]$.

State whether you agree or disagree with this claim. Use evidence from your graph in Fig. 2.1 to support your answer.

 (c) Steps 1 to 4 represent a possible mechanism for the catalysed decomposition of H₂O₂. In this mechanism, the O₂H ligand on one of the complex ions represents the H−O−O⁻ ion and the O represents an oxygen atom.

step 1	$[Fe(H_2O)_6]^{3+}(aq) + H_2O_2(aq) \rightleftharpoons [Fe(H_2O)_5(O_2H)]^{2+}(aq) + H_3O^+(aq)$
step 2	$[Fe(H_2O)_5(O_2H)]^{2+}(aq) \to OH^{-}(aq) + [Fe(H_2O)_5O]^{3+}(aq)$
step 3	$[Fe(H_2O)_5O]^{3+}(aq) + H_2O_2(aq) \rightarrow [Fe(H_2O)_6]^{3+}(aq) + O_2(g)$
step 4	$H_3O^+(aq) + OH^-(aq) \rightarrow 2H_2O(I)$

(i) Perform the test described in Table 2.1 and record your observations in the table. Test and identify any gases produced.

Та	bl	е	2.	1
	~	-	_	

test	observations
Put about 2 cm depth of H_2O_2	
from the bench reagent	
bottle into a test-tube.	
Add about 2 cm depth of	A brown solution was initially observed on adding
FA 4 to the same test-tube	orange $[Fe(H_2O)_6]^{3+}$.
thoroughly	Then vigorous offervescence of a colourless gas
thoroughly.	which relights a glowing
Observe the mixture until no	splint The das is Ω_{2}
further changes are seen	
	An orange/vellow solution was observed at the
	end.
	[2]

(ii) Identify from the mechanism in (c), the complex ions that are responsible for the colour changes you observed in Table 2.1.

(iii) Explain fully how you can tell that the [Fe(H₂O)₆]³⁺ ions are acting as a catalyst in this reaction, using evidence from your observations in Table 2.1 and from the mechanism.

[3]

3 Qualitative Analysis

FA 8 is a solid oxide of an unknown metal **Z**. This oxide has an M_r not exceeding 140.

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[*A*_r of A*l*: 27.0; Ba: 137.3; Ca: 40.1; Cr: 52.0; Cu: 63.5; Fe: 55.8; Mg: 24.3; Mn: 54.9; O: 16.0; Zn: 65.4]

FA 9 is solid potassium ethanedioate, $K_2C_2O_4$.

(a) Perform the tests described in Table 3.1, and record your observations in the table. Test and identify any gases produced.

Table 3.1

test	observations
Add all of the solid FA 9 into a boiling tube. Add 15 cm ³ of FA 7 into this boiling tube.	
Gently warm the boiling tube and stir the mixture using the thermometer with 1 °C divisions, until the temperature of the mixture reaches about 60 °C.	White solid dissolves to give a colourless solution.
Turn off the Bunsen flame.	
Use a spatula to add FA 8 to the mixture, until no further change is seen.	Effervescence/bubbles of gas Gas gives white ppt when bubbled through limewater. Gas is CO ₂ .
When you think that the reaction is complete, stop adding FA 8 .	Black residue observed
Filter the mixture into another boiling tube.	Colourless/pale pink/yellow filtrate
To 1 cm depth of the filtrate in a test tube, add aqueous sodium hydroxide dropwise, until in excess.	Filtrate gives off-white/light brown ppt with NaOH(aq) Ppt is insoluble in excess
	Ppt rapidly turns brown on contact with air

[4]

(b) Explain how you have decided to stop adding **FA 8** at the point you have chosen.

[1]

(c) What is the chemical role of FA 8 in the reaction between FA 8 and FA 9? Support your answer using your observations from Table 3.1.[1] (d) (i) Identify the cation in the filtrate, using your observations from Table 3.1.[1] (ii) Hence, suggest the formula of the oxide of Z in FA 8 and explain your reasoning by referring to your answer in (c) and the information provided on page 10.[2]

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[Total: 9]

4 Planning

When some aqueous ethanoic acid is added to cyclohexane, the ethanoic acid distributes itself in the two immiscible solvents until an equilibrium is set up:

$$CH_3CO_2H(\text{in water}) \Rightarrow CH_3CO_2H(\text{in cyclohexane})$$

The equilibrium constant for this system is called the *partition coefficient*, *K*, which is a constant at a given temperature.

$$K = \frac{[\text{ethanoic acid}] \text{ in cyclohexane}}{[\text{ethanoic acid}] \text{ in water}}$$

To determine the value of K, it is necessary to determine the amount and then the concentration of ethanoic acid in each solvent layer at equilibrium. One way of doing this is described below.

Known volumes of aqueous ethanoic acid and cyclohexane are placed in a separating funnel, shown in Fig. 4.1, and mixed well.

Fig. 4.1

Sufficient time must be provided to allow the following to occur.

- Distribution of the ethanoic acid molecules from the water solvent into the cyclohexane solvent until equilibrium is reached.
- Separation of the mixture into two immiscible aqueous and cyclohexane layers. (The density of cyclohexane is 0.78 g cm⁻³. You may assume that the density of the aqueous layer is the same as that of water, 1.00 g cm⁻³.)

Most of the aqueous layer is then taken out, from which a 10.0 cm³ aliquot is withdrawn for titration.

The experiment is repeated using different volumes of aqueous ethanoic acid, and adding water such that the total volume of the aqueous and cyclohexane layers is kept constant.

Each titration only needs to be done once.

(a) Using the information given, write a plan to collect sufficient data to plot a graph of the equilibrium concentration of ethanoic acid in cyclohexane against that in water, at room temperature.

You are provided with:

- 0.250 mol dm⁻³ aqueous ethanoic acid,
- cyclohexane,
- deionised water,
- 0.100 mol dm⁻³ aqueous sodium hydroxide,
- a separating funnel with capacity 250 cm³,
- other apparatus normally found in a school laboratory.

Your plan should include:

- essential details for the preparation of each equilibrium mixture,
- essential details for the titration of each aqueous aliquot against the sodium hydroxide using thymol blue indicator.

Thymol blue indicator has two pH working ranges as follows.

pH working range	colour in acidic region	colour in basic region
1.2 – 2.8	red	yellow
8.0 - 9.6	yellow	blue

Your first equilibrium mixture should be prepared using 50 cm³ of aqueous ethanoic acid and 50 cm³ of cyclohexane. You may draw a table to show the volumes of aqueous ethanoic acid, deionised water and cyclohexane used to prepare each equilibrium mixture.

You may assume that temperature stays constant throughout your experiment.

.....[5]

Outline how you would calculate the concentration (in mol dm⁻³) of ethanoic acid in each (b) solvent, for equilibrium mixture 1, assuming that the titre volume is $V \text{ cm}^3$.

(c) (i) Sketch on Fig. 4.2, a graph to show the relationship between the equilibrium concentrations of ethanoic acid in the two solvents.

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HWA CHONG INSTITUTION 2022 C2 H2 CHEMISTRY PRELIMINARY EXAM SUGGESTED SOLUTIONS (PAPER 4)

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1 (a) (i) 1m: Fill up Table 1.1 AND both ΔT correctly calculated AND all T & ΔT values to 1 d.p.

1m: Accuracy

1 (a) (ii) 1m: Acid A is monobasic

1m: Explanation based on comparison of ΔT for expt 1 vs. expt 2 AND discussion of limiting/excess reactant and no. of moles of H₂O produced

Sample answer:

		η(OH⁻) /mol	η(A) /mol	η(H⁺) /mol	η(H₂O
					formed) /mol
lf A	expt 1	0.06	0.015	0.015	0.015
monobasic		(excess)	(limiting)		
	expt 2	0.03	0.03	0.03	0.03
		(stoich. amt)		(stoich. amt)	
If A dibasic	expt 1	0.06	0.015	0.03	0.03
		(excess)		(limiting)	
	expt 2	0.03	0.03	0.06	0.03
		(limiting)		(excess)	

A is monobasic. The amount of water formed in experiment 2 is twice that in experiment 1. This means that twice the amount of heat is liberated; as total volume is the same in both experiments, this is reflected in ΔT for experiment 2 (6.2°C) being double that in experiment 1 (3.1 °C). If A were dibasic, the amount of water formed in both experiments is the same, and the ΔT would be the same.

1 (a) (iii) 1m: Calculate heat change q (may use expt 1 or expt 2)

1m: Calculate ΔH_{neut} including – sign

Sample calculation: Heat change q = $60 \times 1.00 \times 4.18 \times 6.2 = 1555 \text{ J}$ n(H₂O) produced = n(NaOH) reacted = $1.50 \times \frac{20}{1000} = 0.03 \text{ mol}$ $\Delta H_{neut} = -\frac{1554.96}{0.03} = -51.8 \text{ kJ mol}^{-1}$

1 (a) (iv) 1m: Δ T doubles

1m: So q doubles but will be divided by double the no. of moles of water produced, therefore no effect on ΔH_{neut}

1 (b) (i) 1m: 20.0 cm³ FA 3 + 40.0 cm³ FA 2, both volumes to 1 d.p. AND Δ T correctly calculated AND student's Δ T within 4.5 – 5.5

(Typical result: $\Delta T_3 = 5.0 \ ^{\circ}C$)

1 (b) (ii) 1m: B is a weak base and compare ΔT for expt 3 (smaller) vs. expt 2

AND some energy is required to fully dissociate B (which was partially dissociated), resulting in less exothermic ΔH_{neut} , although same no. of moles of H₂O produced in expt 3 and expt 2.

- 2 (a) (i) 1m: Headers & units (for both time and titration recordings)
 - Reject 'final volume' 'initial volume' 'amount'
 - This mark is lost if final burette reading < initial or 50.00 used as initial

1m:

- transfer time in min & s to nearest whole number
- t_d correctly calculated to 1 d.p.

1m:

- burette readings, volumes to nearest 0.05 cm³ (2 d.p.)
- correct subtraction between final & initial burette readings

1m:

- transfer time for the first aliquot is within first 3 min of starting the reaction (accept up to 3min 56s = 3.9min)
- last aliquot not exceeding 25 min

1m: 5 sets of results

time		t./min	Final Burette	Initial Burette	Vol of FA5 / cm ³
min	S	ld / mm	reading / cm ³	reading / cm ³	
3	1	3.0	35.10	0.10	35.00
7	0	7.0	24.80	0.00	24.80
12	30	12.5	15.40	0.40	15.00
18	7	18.1	25.20	15.60	9.60
23	0	23.0	31.60	25.20	6.40

2 (a) (ii)

1m:

- Label x & y axes including units
- Plotted points occupy at least ½ of graph grid in both x & y directions
- Reject awkward scale (e.g. 3:10)

1m: Plot all points (±1/2 small square allowance)

1m: Draw best-fit smooth curve and extend it to $t_d = 0.0$ min

1m: Read V₀ correctly (±¹/₂ small square allowance)

gradient =
$$\frac{44.50 - 0}{0 - 14.5}$$
 = -3.0690 \approx -3.07 cm³ min⁻¹

1m: Draw tangent at $t_d = 0.0$ min, tangent line must touch the curve at $t_d = 0.0$

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1m: Read coordinates correctly ($\pm \frac{1}{2}$ small square allowance) AND Calculate gradient, include negative sign

2 (b) (ii) 1m:

$$\frac{-3.0690 \text{ cm}^3 \text{ MnO}_{4^-}}{1 \text{ min}} = \frac{-\frac{3.0690}{1000} \text{ dm}^3 \times 0.0200 \text{ mol dm}^{-3}}{1 \text{ min}}$$
$$= -6.1379 \times 10^{-5}$$
$$\approx -6.14 \times 10^{-5} \text{ mol min}^{-1}$$

2 (b) (iii)
$$2MnO_4^- + 5H_2O_2 + 6H^+ \rightarrow products$$

1m: n(H₂O₂) decomposed per minute =
$$6.1379 \times 10^{-5} \times \frac{5}{2}$$

= 1.5345×10^{-4}
 $\approx 1.53 \times 10^{-4}$ mol

1m:

rate of change of
$$[H_2O_2] = \frac{-1.5345 \times 10^{-4}}{10/1000} = -0.0153 \text{ mol } dm^{-3} \text{ min}^{-1}$$

1m: sf for 1(a)(iii), 2(b) and units for 1(a)(iii)

2 (b) (iv) Volume FA $3/cm^3$ half-life /min 44.50 \rightarrow 22.25 8.0 - 0.0 = 8.022.25 \rightarrow 11.125 16.0 - 8.0 = 8.0

Agree. The half-lives are constant at 8.0 minutes. Since the $t_{1/2}$ are constant, it is a 1st order reaction with respect to H_2O_2 .

1m: Quote at least two half-life values or label on the graph 1m: Conclusion based on whether half-life is (approximately) constant or not

2 (c) (i) A <u>brown solution</u> was initially observed on adding orange $[Fe(H_2O)_6]^{3+}$.

Then vigorous <u>effervescence</u> of a colourless gas which <u>relights a glowing</u> splint. The gas is O_2

An orange/yellow solution was observed at the end.

 $^{1\!\!/_2}$ m for each underlined observation \Rightarrow 2m

- **2** (c) (ii) 1m: Suggests $[Fe(H_2O)_5(O_2H)]^{2+}$ as the brown intermediate (accept $[Fe(H_2O)_5O]^{3+}$) 1m: suggests orange/yellow $[Fe(H_2O)_6]^{3+}$ regenerated
- **2** (c) (iii) 1m: A brown intermediate was formed, showing that $[Fe(H_2O)_6]^{3+}$ participated in the catalysed pathway, as shown in step 1

1m: Vigorous effervescence of oxygen indicated an increase in the rate of reaction

1m: The catalyst was regenerated as seen by the return of the yellow/orange colour at the end and as shown in step 3

3 (a) 1. White solid dissolves to give a colourless solution.

- 2. Effervescence/bubbles of gas
- 3. Gas gives white ppt when bubbled through limewater. Gas is CO₂.
- 4. Black residue
- 5. Colourless/pale pink/yellow filtrate
- 6. Filtrate gives off-white/light brown ppt with NaOH(aq)
- 7. Ppt is insoluble in excess
- 8. Ppt rapidly turns brown on contact with air

1m for every two observations \Rightarrow 4m

- 3 (b) 1m: No more effervescence is observed when FA 8 is added.
- **3** (c) 1m: Oxidising agent, as ethanedioate/ethanedioic acid was oxidised to carbon dioxide.
- 3 (d) (i) 1m: Mn²⁺, as off-white ppt rapidly turns brown on contact with air, insoluble in excess
- **3 (d) (ii)** 1m: MnO₂

1m: FA 8 acted as an oxidising agent, so itself must have been reduced from a higher oxidation state of Mn to Mn^{2+} , and MnO_2 , the oxide with Mn in +4 oxidation state, has $M_r = 86.9$, which is less than 140.

4 (a) There are 10 marking points (M1 – M10)

No. of correct marking points	0–1	2–3	4–5	6–7	8–9	10
No. of marks scored	0	1	2	3	4	5

M1: <u>Separate</u> measuring cylinders (or burettes) to measure volume of <u>each</u> liquid (aqueous ethanoic acid, cyclohexane, deionised water).

M2: Shake the mixture in the separating funnel (or invert the separating funnel a few times to mix the liquids). Then leave the mixture to stand / to equilibrate / to separate into two layers.

M3: Mixture 1 contains 50 cm³ aq. ethanoic acid and 50 cm³ cyclohexane.

AND at least a total of four mixtures stating the volumes of aq. ethanoic acid, cyclohexane and deionised water.

(The top of pg 13 says 'Using the information given'. The bottom of pg 12 says 'using different volumes of aq. ethanoic acid, and adding water such that the total volume of the aq. and cyclohexane layers is kept constant'. So you should stick to 50 cm³ cyclohexane for all mixtures.)

M4: Total volume of aq. layer and cyclohexane layer kept constant.

(M4 is lost if student mixed aq. ethanoic acid and water, never add cyclohexane / mixed water and cyclohexane, never add aq. ethanoic acid.)

M5: Turn the tap of the separating funnel to drain out most of the lower aqueous layer into a beaker.

(M5 is lost if student uses the wrong apparatus e.g. volumetric flask / conical flask to prepare the mixture. Please use separating funnel.)

(Some students poured aq. ethanoic acid/cyclohexane into a beaker/conical flask, then transferred the mixture into the separating funnel (instead of using the separating funnel

<u>directly</u>). If you do this, some liquids will stay in the beaker/conical flask \Rightarrow loss of reagents \Rightarrow M5 lost.)

M6: Pipette out 10.0 cm³ of the aqueous layer from the beaker (after M5). (Reject use of dropping pipette here. The dropping pipette (it's actually just a plastic dropper) was used in Question 2 because the bubbling H_2O_2 makes it difficult to use a 10.0 cm³ or 25.0 cm³ glass pipette.)

M7: Place the 10.0 cm³ aqueous aliquot into a conical flask.

M8: Add 2 or 3 drops of thymol blue to the aq. aliquot.

M9: Titrate the aq. aliquot with aq. NaOH from a burette. AND record initial burette reading and final burette reading.

M10: Titrate until yellow turns green.

(The cyclohexane layer cannot be titrated with NaOH(aq). NaOH is an ionic compound, its OH^- ions have no favorable interactions with the non-polar cyclohexane molecules. So the OH^- ions will not 'enter' the organic solvent to react with the CH_3CO_2H molecules.)

SAMPLE WRITE-UP:

The equilibrium mixtures to be prepared are stated below.

mixture		Volume /cm ³		Titration of 10.0 cm ³ aq. aliquot against NaOH(aq)			
	aq. ethanoic acid	deionised water	cyclohexane	Final burette reading /cm ³	Initial burette reading /cm ³	Volume of NaOH(aq) needed /cm ³	
1	50	0	50				
2	40	10	50				
3	30	20	50				
4	20	30	50				
5	10	40	50				

- 1. Use separate measuring cylinders to measure 50 cm³ of aq. ethanoic acid and 50 cm³ of cyclohexane. Add both liquids into the separating funnel.
- 2. Stopper and shake the separating funnel to mix the liquids. Leave the separating funnel to stand for a few hours.
- 3. Turn the tap of the separating funnel to drain out most of the lower aqueous layer into a beaker.
- 4. Pipette 10.0 cm³ of the aqueous layer, from the beaker in step 3, into a conical flask. Add 2 or 3 drops of thymol blue indicator.
- 5. Titrate the aqueous aliquot with (or against) the NaOH(aq) from a burette until the solution just changes from yellow to green. Record the initial and final burette readings.

- 6. Repeat steps 1 to 2 to prepare the other four mixtures. Use a third measuring cylinder to measure the volume of deionised water. Then repeat steps 3 to 5 for the titration of the aqueous aliquot.
- 4 (b) 1m: n(NaOH) needed for titration 1m: [CH₃CO₂H] in aqueous layer 1m: subtraction working to obtain n(CH₃CO₂H) in cyclohexane layer including scale-upworking for n(CH₃CO₂H) in the aqueous layer 1m: [CH₃CO₂H] in cyclohexane layer

(equilibrium mixture 1: 50 cm³ aq. $CH_3CO_2H + 50$ cm³ cyclohexane) (V cm³ NaOH(aq) needed for titration of 10.0 cm³ aq. layer)

$$n(NaOH) = \frac{V}{1000} \times 0.100 = a \text{ mol}$$

n(CH₃CO₂H) in 10.0 cm³ aq. layer = a mol [CH₃CO₂H] in aq. layer = $\frac{a}{10/1000}$ mol dm⁻³

n(CH₃CO₂H) in 50 cm³ aq. layer = $a \times \frac{50}{10} = b$ mol n(CH₃CO₂H) in the original 50 cm³ aq. acid = $\frac{50}{1000} \times 0.250 = 0.0125$ mol n(CH₃CO₂H) in cyclohexane layer = 0.0125 - b mol

 $[CH_3CO_2H]$ in cyclohexane layer = $\frac{0.0125 - b}{50/1000}$ mol dm⁻³

4 (c) (i)

1m: draw a straight line, positive gradient, joins origin (steepness doesn't matter)

- 4 (c) (ii) 1m: K = gradient of this line
- 4 (d) 1m: Ethanoic acid is a weak acid that <u>ionises partially / dissociates partially in water</u> to give CH₃CO₂⁻ and H⁺ ions. (Reject 'hydrolyses' / 'hydrolysis') or Ethanoic acid molecules may form <u>dimers in the cyclohexane</u> solvent. Therefore, the assumption is not entirely true.