H

NANYANG JUNIOR COLLEGE
JC2 PRACTICAL PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME	

CLASS	1	9				TUTOR					
CENTRE NUMBER	S					INDEX NUMBER					

CHEMISTRY 9729/04

Paper 4 Practical

19 August 2020

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, highlighters, 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 17 and 18.

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(c), (d), (f) [5]		3(a) [8]		**1**	/ 10
1(e) [5]		3(b) [2]		**2**	/ 18
2(a) [7]		4(a)-(c) [6]		**3**	/10
2(b), (c) [6]		4(d)-(g) [6]		**4**	/ 17
2(d), (e) [5]		4(h)-(k) [5]		**Total**	/55

This document consists of **18** printed pages and **2** blank pages.

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

1 Investigation of the extraction of propanoic acid from the aqueous layer by an organic liquid

Propanoic acid has different solubilities in water and in the organic liquid, **FB 6**.

When an aqueous solution of propanoic acid is shaken with **FB 6**, two separate layers, an aqueous layer and an organic layer will be observed. Some of the acid dissolves in the organic layer.

The amount of acid remaining in the aqueous layer can be determined by titration with aqueous sodium hydroxide.

FA 1 is 0.050 mol dm^{-3} sodium hydroxide solution.
FA 2 is 0.200 mol dm^{-3} propanoic acid, C_2H_5COOH.
FB 6 is an organic liquid that does not mix with water.

(a) Preparation of the mixture in Flask A.

1. **Flask A** is a stoppered flask that contains 40 cm^3 of **FB 6**.
2. Use a 50 cm^3 measuring cylinder to place 50 cm^3 of **FA 2** into **Flask A**.
3. Replace the stopper in the flask.
4. Shake the flask vigorously for about 1 minute.
5. **Leave the flask to stand on the workbench for about 10 minutes.**
6. Shake the flask vigorously again for about 1 minute during the 10 minute duration.

While the flask is standing on the workbench, you may move on to the next question.

(b) Titrations

1. Fill the burette with **FA 1**.
2. Ensure the two layers in **Flask A** have separated – this should take no longer than 1 minute after shaking the flask.
3. Pipette 10.0 cm^3 of the **lower** (aqueous) layer into a conical flask. Attach the pipette filler to the pipette before inserting it into the mixture.
4. Replace the stopper in the flask.
5. Titrate the acid in the conical flask with **FA 1**, using thymolphthalein indicator. The end-point is reached when the solution in the flask becomes a permanent pale blue colour.
6. Repeat steps 3 to 5 until consistent titre values are obtained.

(c) **Results**

Record the burette readings and volume of **FA 1** added to the conical flask.

[2]

(d) From your titrations, obtain a suitable volume of **FA 1** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 1** = …………………….. cm^3 [1]

(e) **Calculations**

(i) Use your answer in **(c)** to calculate the volume of **FA 1** needed to react with the acid remaining in **50 cm^3** of the aqueous layer, **after** shaking with the organic liquid.

volume of **FA 1** = ……………….. cm^3 [1]

(ii) It is suggested that shaking the mixture leads to the following equilibrium being established.

$$C_2H_5CO_2H(aq) \rightleftharpoons C_2H_5CO_2H(org)$$

Use the information given and your answer in **(e)(i)** to calculate the equilibrium amount of $C_2H_5CO_2H$(aq) and $C_2H_5CO_2H$(org).

Equilibrium amount of $C_2H_5CO_2H$(aq) = ……………………. mol [1]

Equilibrium amount of $C_2H_5CO_2H$(org) = ……………………. mol [1]

(iii) The expression for the equilibrium constant is as shown below.

$$K_c = \frac{[C_2H_5CO_2H_{(org)}]}{[C_2H_5CO_2H_{(aq)}]}$$

Using the above expression, determine a value for the equilibrium constant, K_c.

K_c = ……………………. [2]

(f) **FB 6** is butan-1-ol. The above experiment is repeated using 2-methylpentane instead of **FB 6**. State and explain the effect on the volume of **FA 1** obtained in **(c)**.

Effect on the volume of **FA 1** ………………………………..……………. [1]

Explanation ……………………………………………………..………………

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[Total: 10]

2 Determination of the concentration of sulfuric acid, FA 4 and its enthalpy change of reaction with aqueous sodium hydroxide.

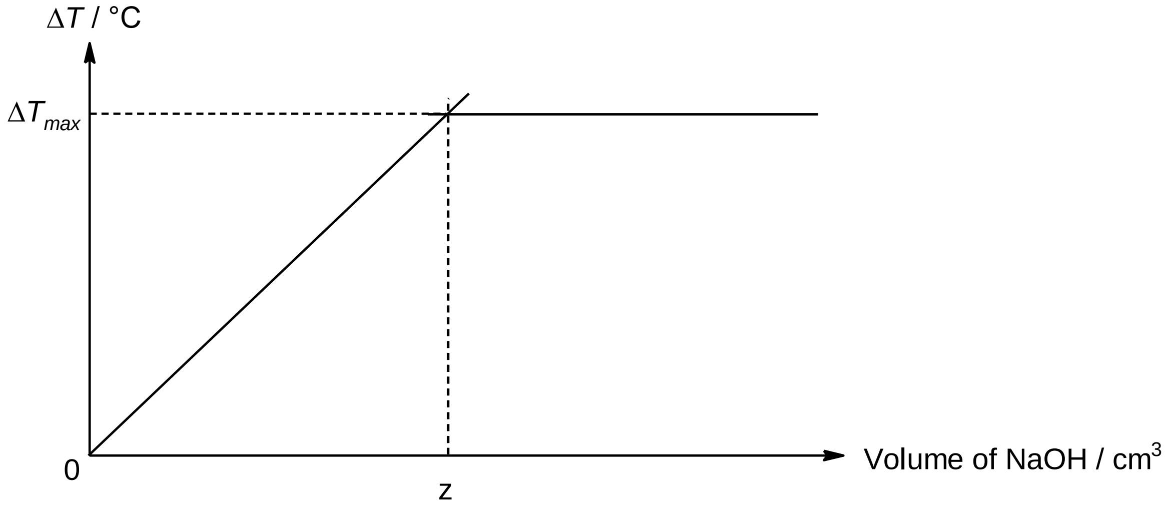
The enthalpy change of neutralisation, ΔH_n, is the enthalpy change when one mole of water is formed during a neutralisation reaction as shown in the equation.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

The enthalpy change of neutralisation between an acid and an alkali can be determined by adding various volumes of acid and alkali and measuring the change in temperature. It is possible to make use of this fact to determine the equivalence point of a neutralisation reaction without the use of an indicator. The equivalence point is the point where H^+(aq) from the acid and OH^-(aq) from the base are present in equal molar amounts.

Using this process, you are to perform a series of experiments where different volumes of alkali are added to a fixed volume of 25 cm^3 of sulfuric acid while keeping the total volume of the solution at 100 cm^3 by adding appropriate volumes of water and determining the temperature change, ΔT, for each experiment.

You will then plot a graph of ΔT against volume of NaOH used.



You are to determine experimentally

- z, the exact volume of NaOH which reacts with 25 cm^3 of H_2SO_4
- ΔT_{max}

You are provided with the following reagents:

- **FA 3** is 1.50 mol dm^{-3} NaOH
- **FA 4** is H_2SO_4 of approximate concentration 1.0 mol dm^{-3}
- deionised water

(a) Method

Experiment 1

1. Using a 100 cm^3 measuring cylinder, place 25 cm^3 of **FA 4** and 65 cm^3 of deionised water into the styrofoam cup provided. Place the cup inside a 250 cm^3 glass beaker to prevent it from tipping over.

2. Measure and record the initial temperature of the mixture of **FA 4** and water.

3. Using another 100 cm^3 measuring cylinder**,** measure 10 cm^3 of **FA 3**. Measure and record the initial temperature of **FA 3**.

4. Calculate and record the average initial temperature.

5. Add **FA 3** quickly from the second measuring cylinder into the cup. Cover the cup with lid provided and stir the mixture immediately. Measure the maximum temperature obtained. Record this temperature.

6. Calculate and record the change in temperature, ΔT.

7. Wash and carefully dry the styrofoam cup.

Experiment 2

Repeat experiment **1** above using 45 cm^3 of water and 30 cm^3 of **FA 3** instead.

Experiments 3, 4, 5 and 6

35 cm^3 of **FA 3** is a rough value of z. You are to perform four more experiments, each with a different volume of **FA 3** added to 25 cm^3 **FA 4** in order to plot a graph of the form shown on page 5 and to obtain an exact value for z.

Remember

- You should **not** use more than 60 cm^3 of **FA 3** for any experiments.
- You already have data for 10 cm^3 of **FA 3** and 30 cm^3 of **FA 3** added.
- You should record all your results in a single table.

Results

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(b) Use the grid below to plot a graph of ΔT against volume of **FA 3** added.

I	
II	

[2]

(c) Draw two straight lines through the plotted points to find z, the volume of **FA 3** that just reacts with 25 cm^3 of **FA 4** and the maximum temperature change, ΔT_{max}.

z, the volume of **FA 3** = …………………….

maximum temperature change, $\Delta \boldsymbol{T}_{max}$ = …………………….

Teacher's z	
diff.	
Teacher's $\Delta \boldsymbol{T}_{max}$	
diff.	

[4]

(d) Use your results from part **(c)** to calculate:

(i) the concentration of H_2SO_4 in the **FA 4** solution and

(ii) a value for the enthalpy change, ΔH_1 of the following reaction between H_2SO_4 and NaOH.

$$H_2SO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) \qquad \Delta H_1$$

You should assume that the specific heat capacity of the final solution is 4.18 J g^{-1} K^{-1}, and that its density is 1.00 g cm^{-3}.

concentration of H_2SO_4 in the **FA 4** solution = ………………….. [1]

ΔH_1 = ………………….. [2]

(e) Consider the shapes of your graph lines.

(i) Explain the shape of your graph line **before** the equivalence point.

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.... [1]

(ii) Explain the shape of your graph line **after** the equivalence point.

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.... [1]

[Total: 18]

3 Planning

The solubility of calcium hydroxide, at a particular temperature, is defined as the maximum mass of calcium hydroxide that can dissolve in and just saturate 100 g of solvent at that temperature. It can be represented by the following equation:

$$\text{Solubility} = \frac{\text{mass of calcium hydroxide (g)}}{\text{100 g of solvent}}$$

A **saturated** solution is one in which no more solid can dissolve at a particular temperature.

When calcium hydroxide dissolves in water, the following equilibrium is established.

$$Ca(OH)_2(s) + aq \rightleftharpoons Ca^{2+}(aq) + 2OH^{-}(aq) \text{ -------------------- (1)}$$

Calcium hydroxide also dissolves in an aqueous solution of sodium hydroxide.

Calcium hydroxide undergoes decomposition at about 512 ^{o}C.

You are to plan an experiment to investigate how the **solubility** of calcium hydroxide in an aqueous solution of sodium hydroxide depends on the concentration of the sodium hydroxide.

You are to design a two-part laboratory experiment to investigate your prediction in **(a)**.

(a) Part 1 – solubility of calcium hydroxide in water at 10 ^{o}C

In addition to the standard apparatus present in a laboratory, you are provided with the following materials.

150 cm^3 distilled water
5 g solid calcium hydroxide, $Ca(OH)_2$
ice cubes in a tub
thermometer
IR lamp

Give a step-by-step description of how you would

- prepare a saturated solution using the distilled water given,
- control the temperature of the experiment,
- separate the saturated solution from undissolved solid,
- obtain the mass of calcium hydroxide and mass of water in the saturated solution,
- determine the solubility of calcium hydroxide from the experimental results.

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(b) Part 2 – solubility of calcium hydroxide in an aqueous solution of sodium hydroxide

In addition to other standard apparatus present in a laboratory, you are provided with the following materials.

0.5 mol dm^{-3} NaOH
1.5 mol dm^{-3} NaOH from Question **2**

Give a step-by-step description of how you would prepare four solutions of sodium hydroxide with concentrations ranging from 0.25 to 1.5 mol dm^{-3}. These solutions could then be used to investigate the solubility of calcium hydroxide in different concentrations of sodium hydroxide solutions.

Your plan should include details of the following.

- the volumes of the given sodium hydroxide solutions used to prepare the four solutions of sodium hydroxide of concentrations ranging from 0.25 to 1.5 mol dm^{-3},
- the apparatus and method you would use in their preparation.

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[Total: 10]

4 Investigation of a gemstone

Spinel minerals form crystals that are often used as gemstones. The general formula of a spinel mineral is $\mathbf{AB_2O_4}$, where **A** and **B** are different metal ions. The presence of different combinations of metal ions accounts for the range of colours found in these gemstones. One example of a spinel gemstone is *hercynite*, which is blue-green in colour.

As they are essentially composed of metal oxides, spinel crystals may be reacted with strong acids to form solutions containing salts of the metal ions present.

The **FA 5** solution you are supplied with is a mixture of two salts. It is intended to duplicate the solution that could have been produced by reacting hercynite crystals with a strong mineral acid.

FA 5 is a solution containing the metal ions, **A** and **B**
FA 6 is sodium carbonate, Na_2CO_3

You are to determine the identities of the metal ions, **A** and **B**, present in **FA 5** and explain the observations you make in terms of the chemistry involved.

Carry out the following experiments. Carefully record your observations. Test any gases produced.

The volumes given below are approximate and should be estimated rather than measured, unless otherwise stated.

	test	observations
(a)	Test the **FA 5** solution using Universal Indicator paper.	
(b)	1. Using a measuring cylinder, transfer 10 cm^3 of **FA 5** to a clean boiling tube. 2. Using another measuring cylinder, measure out 10 cm^3 of **FA 6**. 3. Use a dropping pipette to transfer 1.0 cm^3 of **FA 6** from the measuring cylinder to the boiling tube. 4. Mix the contents of the boiling tube thoroughly. 5. Repeat points 3 and 4 until the measuring cylinder is empty. 6. Swirl and leave the mixture to stand for 5 minutes.	

(c)(i)	Place about 2 cm depth of **FA 5** into a test-tube. Carefully add sodium hydroxide, dropwise with shaking, until no further changes are seen. Swirl and filter the mixture, collecting the filtrate in a test-tube. The filtrate is **FA 7** which should be put to one side for use in **(ii)**. Wash the residue thoroughly with deionised water. Discard the washings. The residue is **FA 8**. Retain the residue for use in **(iii)**.	
(ii)	To the filtrate, **FA 7** from **(c)(i)**, carefully add hydrochloric acid, dropwise *(do not shake the mixture while adding the* ***HCl****)*, until no further change is seen.	
	Note: When aqueous potassium manganate(VII) is added in **(c)(iii)**, the endpoint is a permanent **pale** pink colour. Use the same dropping pipette and record the number of drops of aqueous potassium manganate(VII) you added to reach the endpoint.	
(iii)	Transfer **FA 8** into a clean test-tube. Carefully add hydrochloric acid, a few drops at a time with shaking, until no further changes are seen. Add aqueous potassium manganate(VII), dropwise with shaking, until the endpoint is reached.	

Obs points
marks

[6]

(d) Metal ions **A** and **B** can be found in the Qualitative Analysis Notes. By considering the general formula of the spinel minerals, AB_2O_4, suggest the likely oxidation number of the metal ions, **A** and **B**.

metal ion **A**: ……………….. metal ion **B**: ………………..

[1]

(e) From the observations obtained in **(a)**, suggest the nature of **FA 5**.

Metal ion **B** is responsible for the nature of **FA 5**. It is formed from a Period 3 element. Suggest the likely identity of metal ion **B**.

nature of **FA 5**: ………………………………………………………………….... [1]

metal ion **B**: ……………………………………………………………….…. [1]

(f) Consider your observations in **(b)** and your answer in **(e)**, suggest an explanation for the observations in **(b)** in terms of the chemistry involved. Your answer should include the identification of any gas or precipitate produced in **4(b)**.

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(g) Consider your answers in **(d)** to **(f)**, identify the metal-containing complex present in **FA 7** in **(c)(i)**.

metal-containing complex present in **FA 7**: ……………………………….….. [1]

(h) Use Le Chatelier's Principle to explain your observations in **(c)(ii)**.

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(i) Deduce the type of reaction occurring in **(c)(iii)** when HCl(aq) was added.
Write an equation, including state symbols in your answer.

Type of reaction: ……………………………………................................... [1]

Equation: ……………………………………………………………….................. [1]

(j) **FA 5** is intended to duplicate a solution that could have been produced by dissolving hercynite in a strong acid.
Suggest a formula for hercynite.

……………………………………………………………………………………. [1]

[Total: 17]

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, Cl^-(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