Class	Adm No

Candidate Name: ____________________

Shift
Laboratory

H

2018 Preliminary Exams
Pre-University 3

H2 CHEMISTRY **9729/04**

Paper 4 Practical

10th Sept 2018

2 hour 30 mins

Candidates answer on the Question paper.

READ THESE INSTRUCTIONS FIRST

Do not turn over this question paper until you are told to do so

Write your name, class and admission number on all the work you hand in.

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 at the back of the Question Paper.

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.

Question	1	2	3	4	Total
Marks	26	13	6	10	55

1 Investigation of acid-base titrations involving sodium hydrogen carbonate

According to the Arrhenius theory of acids and bases, an acid produces H^+(aq) ions and a base produces OH^-(aq) ions. The reaction of these two ions to form water molecules is known as acid-base neutralisation.

The equation for this neutralisation reaction is given below, and strong acid–strong base reactions are known to have an enthalpy change of neutralisation (ΔH_{neu}) of approximately –57.1 kJ mol^{-1}.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l) \qquad \Delta H_{neu} = -57.1 \text{ kJ mol}^{-1}$$

However, the ΔH_{neu} for weak acid–strong base reactions are known to differ. Sodium hydrogen carbonate, $NaHCO_3$, is an example of a weak acid.

FA 1 is 1.8 mol dm^{-3} sodium hydrogen carbonate, $NaHCO_3$.
FA 2 is sodium hydroxide, NaOH, of concentration between 0.9 – 1.2 mol dm^{-3}.

$$NaHCO_3 + NaOH \rightarrow Na_2CO_3 + H_2O \qquad \Delta H_{neu}'$$

As the precise concentration of **FA 2** is unknown, determination of $\Delta H_{neu}'$ can be done using a thermometric titration to simultaneously determine both the concentration of **FA 2** as well as $\Delta H_{neu}'$. Thermometric titration is a technique whereby equivalence points of a reaction can be located by observing temperature changes, hence eliminating the need for an indicator.

In **1(a)**, you will perform a weak acid–strong base thermometric titration. The data from this titration will be used to determine:

- the titration value at equivalence point, V_{eq},
- the precise concentration of **FA 2**, [NaOH],
- the maximum temperature change, ΔT_{max},
- the enthalpy change of neutralisation, $\Delta H_{neu}'$.

For Examiners' Use

(a) Determination of V_{eq} and ΔH_{neu}' using thermometric titration

For this experiment, you will need to measure the **maximum temperature** of the reaction mixture when specified volumes of **FA 1** have been added.

In an appropriate format in the space provided on the **next page**, prepare a table to record your results. Record all values of temperature, *T*, to 0.1°C, and each total volume of **FA 1** added.

Note: You should aim to perform each subsequent addition of **FA 1** quickly.

1. Fill a burette with **FA 1**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 2** into a Styrofoam cup. Place this cup inside a second Styrofoam cup, which is placed in a 250 cm^3 glass beaker.
3. Stir and measure the temperature of this **FA 2**. Record this temperature.
4. Add 2.00 cm^3 of **FA 1** from the burette to the **FA 2** in the Styrofoam cup.
5. Using the thermometer, stir the mixture thoroughly and record the maximum temperature reached and the volume of **FA 1** added.
6. Repeat steps **4** and **5** until a total volume of 30.00 cm^3 of **FA 1** has been added.

Results

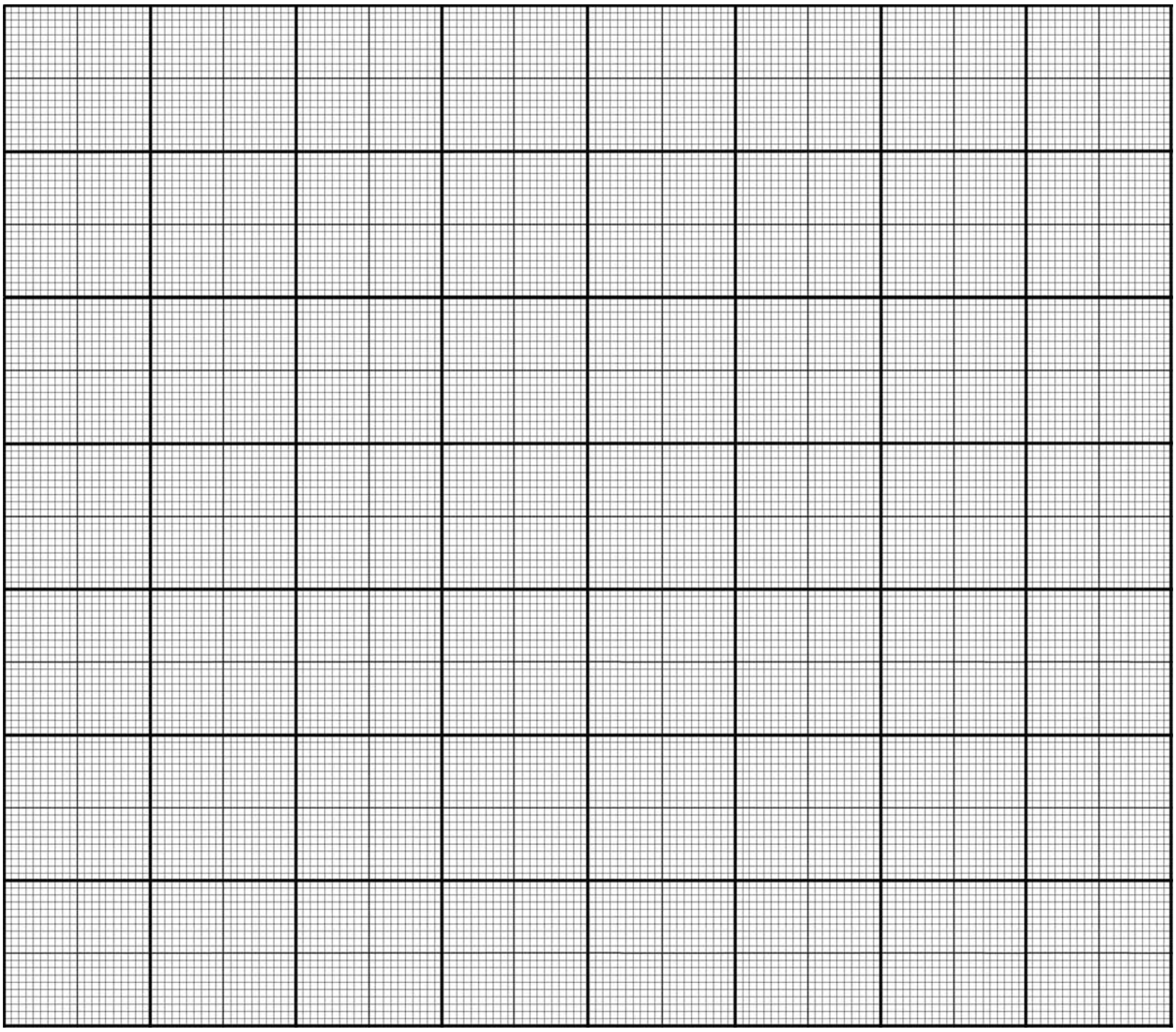
M1

M2

[2]

(i) Plot a graph of temperature, *T*, on the *y*-axis, against volume of **FA 1** added, on the *x*-axis on the grid in **Fig. 1.1**.

The temperature axis should allow you to include a point at least 1.5 °C greater than the maximum temperature recorded.



M3

M4

M5

Fig. 1.1

Draw **two** most appropriate best-fit lines in **Fig. 1.1**, taking into account all of your plotted points.

Extrapolate (extend) these two best-fit lines until they cross each other. [3]

For Examiners' Use

(ii) From your graph in **Fig. 1.1**, determine:

- the titre at equivalence point, V_{eq},
- the maximum temperature reached, T_{max},
- the maximum temperature change, ΔT_{max}.

On your graph, show clearly how you obtained these values.

V_{eq} = ... cm^3

T_{max} = ... °C **M6**

ΔT_{max} = ... °C **M7**

M8

[3]

(iii) Determine the concentration of NaOH, [NaOH], in **FA 2**.

M9

[NaOH] in **FA 2** = ... [1]

(iv) Determine the enthalpy change of neutralisation, ΔH_{neu}'.

$$NaHCO_3 + NaOH \rightarrow Na_2CO_3 + H_2O \qquad \Delta H_{neu}'$$

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

M10

M11

ΔH_{neu}' = ... [3] **M12**

(v) Comment on your value of ΔH_{neu}' obtained compared to ΔH_{neu} = –57.1 kJ mol^{-1}.

....

....[1] **M13**

(vi) From your graph in **Fig. 1.1**, explain the shape of your best-fit line **before** equivalence point.

....

....[1] **M14**

(b) Determination of titration value at equivalence point, V_{eq}', using 'regular' titration

For Examiners' Use

FA 3 is hydrochloric acid, HC*l*, of unknown concentration.

Sodium hydrogen carbonate, $NaHCO_3$, is also able to act as a weak base. In **1(a)**, $NaHCO_3$ acts as the acid while in **1(b)**, $NaHCO_3$ is acts as the base.

For this experiment, you will titrate **FA 3** against **FA 1** to determine the titration value at equivalence point, V_{eq}', using methyl orange as the indicator.

(i) The use of thermometric titration in **1(a)** eliminated the need for an indicator as the equivalence point was located by observing temperature changes. 'Regular' acid-base titrations however, require the use of an indicator.

Explain why an indicator is required for 'regular' acid-base titrations (such as in **1(b)**).

....

....[1] **M15**

Titration of FA 3 against FA 1

1. Fill the burette with **FA 1**.
2. Using a pipette, transfer 25.0 cm^3 of **FA 3** into a conical flask.
3. Add 2 – 3 drops of methyl orange indicator into the same conical flask.
4. Run **FA 1** from the burette into this flask until end-point is reached.
5. Record your titration results in the space provided. Make certain that your recorded results show the precision of your working.
6. Repeat points **1** to **5** as necessary until consistent results are obtained.

Results

M16

M17

M18

[3]

(ii) From your titrations, obtain a suitable volume of **FA 1** to be used in your calculations (titre at equivalence point, V_{eq}'). Show clearly how you obtained this value.

V_{eq}' = ... cm^3 [1] **M19**

For Examiners' Use

(iii) Determine the concentration of HC*l*, [HC*l*], in **FA 3**.

M20

[HC*l*] in **FA 3** = ……………………….. [1]

(iv) Suggest if the use of more drops of methyl orange indicator is appropriate for this titration.

…………………………………………………………………………………………………

M21

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

(c) Planning

The same 'regular' titration in **1(b)** can also be performed using an alternative method involving a pH meter, which is an instrument that can measure the pH of water-based solutions at any one instance of time.

Usage of a pH meter is simple as a digital pH reading will be displayed upon placing it into the solution of interest. However, it is known that most pH meters have to be calibrated (correlate to a standard) before each experiment to ensure the accuracy of measurements. For this titration, the standard chosen for calibration is usually a buffer solution of pH ≈ 4.0.

Plotting pH against titration volume, *V*, will give a pH curve that enables the determination of titration value at equivalence point, V_{eq}''.

Plan an experiment to determine the titration value at equivalence point, V_{eq}'', for the titration of **FA 3** against **FA 1** using a pH meter.

You may assume that you are provided with

- a pH meter,
- **FA 1** and **FA 3**,
- 1.0 mol dm^{-3} of ethanoic acid, CH_3COOH ($K_a = 1.8 \times 10^{-5}$),
- 1.0 mol dm^{-3} of sodium ethanoate solution, $CH_3COO^{-}Na^{+}$,
- the glassware and equipment normally found in a school or college laboratory.

In your plan you should include brief details of

- the preparation of a suitable standard solution for calibration of the pH meter,
- the procedure that you would follow,
- the measurements you would take,
- an outline of how you would use your results to determine V_{eq}''.

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....

....

For Examiners' Use

[5]

[Total: 26]

M22

M23

M24

M25

M26

For Examiners' Use

2 Determination of the major component in a solid mixture

Sodium hydrogen carbonate, $NaHCO_3$, also commonly known as *baking soda*, is a white crystalline solid primarily used in baking as a raising agent.

To increase the strength of *baking soda* as a raising agent, cream of tartar (a dry acid) is mixed with sodium hydrogen carbonate. This mixture is known as *baking powder*.

FA 4 is a sample of *baking powder*.

In this experiment, you will determine if sodium hydrogen carbonate is the major component, by mass, of the mixture **FA 4**.

(a) Thermal decomposition of *baking powder*

You may assume that the cream of tartar in **FA 4** is inert and does not decompose when heated.

1. Weigh and record the mass of an empty boiling-tube.
2. Transfer approximately 2 g of **FA 4** into the weighed boiling-tube. Reweigh and record the mass of the boiling-tube and **FA 4**.
3. Gently heat the **FA 4** in the boiling-tube for 2 minutes, then heat strongly for a further 2 minutes. **Take care not to lose any solid from the tube during heating.**
4. Warm the upper parts of the boiling-tube to evaporate any water that may have condensed while heating the solid.
5. Place the hot tube on the test-tube rack and leave to cool. **You are advised to continue with part 2(c) or to start another question while the tube cools.**
6. When cool, reweigh the boiling-tube and the residual solid.
7. Reheat, cool and reweigh the tube until decomposition is complete.

In an appropriate form in the space below, record all your balance readings, the mass of **FA 4** heated, the mass of residual solid, and the mass lost on heating.

Results

M27

M28

M29

M30

M31

[5]

For Examiners' Use

The thermal decomposition of $NaHCO_3$ produces Na_2CO_3 and two gases at the temperature of decomposition, CO_2 and H_2O.

$$2NaHCO_3(s) \rightarrow Na_2CO_3(s) + CO_2(g) + H_2O(g)$$

(i) By finding the average M_r of the two gases produced, use your results in **(a)** calculate the **total** amount of gases lost upon complete decomposition of **FA 4**.

[A_r: C, 12.0; H, 1.0; Na, 23.0; O,16.0]

total amount of gases lost = …………………… mol [1] M32

(ii) Taking into account the mole ratio of gases in the decomposition equation given, calculate the amount of CO_2(g) lost upon complete decomposition of **FA 4**.

M33

amount of CO_2(g) lost = …………………… mol [1]

(iii) Hence, calculate the mass of $NaHCO_3$ in the sample of **FA 4** heated.

M34

mass of $NaHCO_3$ = ……………………… g [1]

(iv) By means of calculation or otherwise, justify if $NaHCO_3$ is the major component, by mass, of **FA 4**.

…………………………………………………………………………………………………

…………………………………………………………………………………………….. [1] M35

For Examiners' Use

(b) **Do not carry out your suggestions.**

Suggest two ways in which you could show that cream of tartar does not decompose on heating.

(i) ....

....

(ii) **M36**

.... [2] **M37**

(c) A student is asked to weigh, with maximum precision, a solid.

Three balances are available.

- Balance A, reading to 1 decimal place,
- Balance B, reading to 2 decimal places,
- Balance C, reading to 3 decimal places.

> Balance readings can be treated similarly to burette readings.
>
> For example, the smallest division on a burette is 0.1 cm^3.
> The maximum error in a single burette reading is ±0.05 cm^3.

Complete the following table.

balance	maximum error for a single balance reading /g	maximum % error when weighing:
A	±	8.0 g of solid =
B	±	4.00 g of solid =
C	±	0.400 g of solid =

M38

M39

[2]

[Total: 13]

For Examiners' Use

3 Qualitative Analysis of an unknown double salt

Double salts are salts that contain more than one cation or anion, and are synthesised by crystallising a solution containing the different ions.

FA 5 is a double salt which contains **two cations** and **one anion**.

Empty out the **FA 5** provided into a 50 cm^3 beaker. To this beaker, add 10 cm^3 of water and stir to dissolve as much of the solid as possible.

This solution will be referred to as '**FA 5** solution'.

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

Table 3.1

tests		observations
1.	Add about 1 cm depth of **FA 5** solution to a boiling-tube, followed by NaOH(aq) dropwise, until excess (a further 2 cm depth). Warm the solution gently.	
2.	Add about 2 cm depth of **FA 5** solution to a test-tube. To this test-tube, add about half a spatula of zinc powder. Observe the mixture until no further changes are seen.	
3.	Add about 1 cm depth of **FA 5** solution into a test-tube. To this test-tube, add barium nitrate dropwise.	

M40

M41

M42

[4] M43

(b) Based on your observations in **3(a)**, deduce the identities of the ions present in **FA 5**.

M44

cation 1: ………… cation 2: ………… anion: ………… [1]

(c) Hence, suggest a possible chemical formula of the double salt **FA 5**.

M45

chemical formula of **FA 5**: …………………… [1]

[Total: 6]

4 Planning

For Examiners' Use

The condenser is an apparatus frequently used in organic synthesis. It comprises of concentric glass tubes, an inner one through which hot gases can pass through, and an outer one through which a cool fluid can pass through.

Use of the condenser is required for the organic synthesis of aldehydes and carboxylic acids from alcohols. The product of the reaction between potassium dichromate, $K_2Cr_2O_7$, and primary alcohols can either be an aldehyde or a carboxylic acid depending on how the condenser is orientated.

(a) Some organic synthesis procedures require *heating under reflux*.

Explain the role of the condenser in such procedures.

…… **M46**

……………………………………………………………………………………………………….. [2] **M47**

(b) Plan an experiment to synthesise **propanal** (boiling point: 20 °C) from **propan-1-ol** (boiling point: 97 °C).

You may assume that you are provided with

- propan-1-ol, $CH_3CH_2CH_2OH$,
- potassium dichromate, $K_2Cr_2O_7$,
- commonly used organic chemicals
- the glassware and equipment normally found in a school or college laboratory.

In your plan you should include

- the reactants and conditions that you would use,
- a well-labelled diagram of the set-up that you would use,
- the procedure that you would follow and the safety precautions taken,
- how you would check the purity of your product.

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For Examiners' Use

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.... M48

.... M49

.... M50

.... M51

....[4]

(c) Suggest appropriate modifications to your plan to synthesise **propanoic acid** (boiling point: 141 °C) from propan-1-ol instead of propanal.

You may wish to use diagrams to complement your answer.

....

....

.... M52

.... M53

....[2]

(d) Give an explanation for any **two** modifications you have made in **(c)**.

....

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.... M54

.... M55

....[2]

[Total: 10]

End of Paper 4

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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

(b) Reactions of anions

ions	*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 Al foil
nitrite, $NO_2^-(aq)$	NH_3 liberated on heating with $OH^-(aq)$ and Al 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) Test for gases

ions	*reaction*
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_2	black solid / purple gas	brown	purple