NATIONAL JUNIOR COLLEGE
SH 2 Year − End Practical Examination
Higher 2

CANDIDATE NAME	

SUBJECT CLASS		REGISTRATION NUMBER	

CHEMISTRY 9729/04

Paper 4 Practical **Wednesday 28 August 2024**

Candidate answer on the Question paper. **2 hours 30 minutes**

READ THESE INSTRUCTIONS FIRST

Write your identification number and name.
Give details of the practical shift and laboratory where appropriate, in the boxes provided.
Write in 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 21 and 22.

The number of marks is given in brackets [ ] at the end of each question or part question.

Shift
Laboratory

For Examiner's use	
1	/ 14
2	/ 20
3	/ 12
4	/ 9
Total	/ 55

This paper consists of **22** printed pages including this cover page.

1 Determination of a value for an enthalpy change of solution Via Hess's Law.

FA 1 is solid sodium hydrogencarbonate, $NaHCO_3$.
FA 2 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

Sodium hydrogencarbonate dissolves in water according to equation 1.

equation 1 $NaHCO_3(s) + aq \rightarrow Na^+(aq) + HCO_3^-(aq)$ ΔH_1

Both solid and aqueous sodium hydrogencarbonate react with sulfuric acid.

equation 2 $2NaHCO_3(s) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$ ΔH_2

equation 3 $2NaHCO_3(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2CO_2(g)$ ΔH_3

In this question, you will perform an experiment to determine a value for ΔH_2. You will use data provided to calculate ΔH_3 and hence a value for ΔH_1.

(a) Determination of the molar enthalpy change of reaction, ΔH_2

In this experiment, you will determine the maximum temperature change when a known mass of solid sodium hydrogencarbonate, **FA 1**, reacts with excess dilute sulfuric acid, **FA 2**.

In an appropriate format in the space provided on page 3, prepare tables in which to record for your experiment:

- all weighings to an appropriate level of precision,
- all values of temperature, T, to an appropriate level of precision.

Procedure

1. Weigh the capped bottle containing **FA 1**. Record this mass.

2. Place one polystyrene cup inside another polystyrene cup and place both in a glass beaker.

3. Use a 50 cm^3 measuring cylinder to transfer 30.0 cm^3 of **FA 2** into the polystyrene cup.

4. Stir the **FA 2** in the polystyrene cup with the thermometer. Read and record its initial temperature, T_i.

5. Slip the thermometer through the lid. Carefully transfer all the solid **FA 1** in the bottle to the **FA 2** in the polystyrene cup, in small portions, to avoid too much frothing. Secure the lid onto the cup.

6. Use the thermometer to stir the mixture. Observe the temperature until it shows the maximum change from the initial temperature. Record this temperature, T_m.

7. Reweigh the empty capped bottle. Record this mass.

Determine the maximum temperature change, ΔT, and the mass of **FA 1** used.

Results

[5]

(b) In the following calculations, you should assume that the specific heat capacity of the solution is 4.18 J g^{-1} K^{-1}, and the density of the solution is 1.00 g cm^{-3}.

(i) Use your results from **1(a)** to calculate the heat change for your experiment.

heat change = ………………………………………. [1]

(ii) Hence, determine a value for ΔH_2.

Include the sign of ΔH_2 in your answer.

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

ΔH_2 = ………………………………………. [1]

The results of an experiment where a solution of 0.690 mol dm^{-3} aqueous sodium hydrogencarbonate, $NaHCO_3(aq)$ was reacted completely with an excess of dilute sulfuric acid, **FA 2**, are shown in Table 1.1.

Table 1.1

volume of $NaHCO_3(aq)$ used / cm^3	50.0
initial temperature of $NaHCO_3(aq)$ / °C	27.6
volume of **FA 2** used / cm^3	25.0
initial temperature of **FA 2** / °C	31.2
minimum temperature / °C	28.4

(iii) Use the results given in Table 1.1 and the formula below to calculate the weighted average initial temperature, T_{av}, of the reaction mixture.

The formula for T_{av} is given as

$$T_{av} = \frac{(\text{vol. of } \textbf{FA 2} \times \text{initial temp. of } \textbf{FA 2}) + (\text{vol. of } NaHCO_3 \times \text{initial temp. of } NaHCO_3)}{\text{total volume of reaction mixture}}$$

T_{av} = ... [1]

(iv) Hence, calculate a value for ΔH_3.

ΔH_3 = ... [4]

(c) Use your answers from **1(b)(ii)** and **1(b)(iv)** to calculate a value for ΔH_1 for the reaction shown in equation 1.

If you are not able to determine a value for **1(b)(ii)** and/or **1(b)(iv)**, you may use x and y to represent the respective enthalpy changes and proceed with this part of the question.

ΔH_1 = ... [2]

[Total: 14]

2 To determine the order of reaction with respect to the concentration of iodine in the iodination of propanone reaction

FA 2 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.
FA 3 is 1.00 mol dm^{-3} propanone, CH_3COCH_3.
FA 4 is an aqueous solution of iodine, I_2.
FA 5 is 0.0100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 6 is 0.50 mol dm^{-3} sodium hydrogencarbonate, $NaHCO_3$.

You are also provided with a starch indicator.

The equation in reaction 1 represents the reaction between CH_3COCH_3 and I_2.

reaction 1: $CH_3COCH_3(aq) + I_2(aq) \xrightarrow{H^+} CH_3COCH_2I(aq) + HI(aq)$

This reaction is first order with respect to both CH_3COCH_3 and H^+ ions.

You are to investigate the order of reaction with respect to I_2.

A reaction mixture containing an acidified solution of CH_3COCH_3 and I_2 is first prepared. At timed intervals, aliquots (portions) of this reaction mixture will be removed and quenched using excess $NaHCO_3$.

It is **not** essential that you complete the titration of one aliquot before extracting the next one from the reaction mixture.

The remaining amount of I_2 at different times can then be determined by titration against $Na_2S_2O_3$. $Na_2S_2O_3$ reacts with I_2 as shown in the equation in reaction 2.

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

The required order of reaction can be obtained by graphical analysis of your results.

(a) Preparing and titration of the reaction mixture

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

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

You should aim to transfer your first aliquot approximately four minutes after starting the reaction.

You should aim **not** to exceed a maximum reaction time of 20 minutes for this experiment.

In an appropriate format in the space provided on page 8, 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 s then t_d = 4 min + 33/60 min = 4.6 min,
- the burette readings and the volume of **FA 5** added.

Question 2 continues on the next page.

Safety:
Propanone is flammable. Transfer your titrated solutions into the **waste** bottle for later disposal. Keep this bottle stoppered when not in use.

Keep the conical flask labelled **reaction mixture** stoppered except when removing aliquots.

1. Fill a burette with **FA 5**.

2. Using a 25 cm^3 measuring cylinder, add the following to a 100 cm^3 beaker.
    - 25.0 cm^3 of **FA 2**
    - 25.0 cm^3 of **FA 3**

3. Using a 100 cm^3 measuring cylinder, transfer 50.0 cm^3 of **FA 4** into the 250 cm^3 conical flask, labelled **reaction mixture.**

4. Pour the contents of the 100 cm^3 beaker into this 250 cm^3 conical flask. Start the stopwatch, **insert the stopper** and swirl the mixture thoroughly to mix its contents.

5. Using a 10 cm^3 measuring cylinder, measure 10.0 cm^3 of **FA 6** into a second conical flask.

6. At approximately 4 minutes, using a 10.0 cm^3 pipette, remove a 10.0 cm^3 aliquot of the reaction mixture. **Immediately** transfer this aliquot into the second conical flask containing **FA 6** and swirl the mixture thoroughly.

    Note the time of transfer, *t*, to the nearest second, when half of the reaction mixture has been dispensed from the pipette. Replace the stopper in the reaction flask.

7. Titrate the iodine in the second conical flask with **FA 5**. When the colour of the solution turns pale yellow, add about 1 cm^3 of starch indicator. The solution will turn blue–black. The end–point is reached when the blue–black colour just disappears. Record your results.

8. Empty the contents of this conical flask into the waste bottle. Wash this conical flask thoroughly with water.

9. Repeat steps 5 to 8 until a total of **five** aliquots have been titrated and their results recorded.

Results

[3]

(b) **(i)** On the grid in Fig. 2.1, plot a graph of the volume of **FA 5** added, on the *y*–axis, against decimal time, t_d, on the *x*–axis.

Draw the best–fit line taking into account all of your plotted points. Extrapolate (extend) this line to t_d = 0.0 min.

Fig. 2.1

[4]

(ii) Deduce the order of reaction with respect to the [I_2] in reaction 1. Explain your answer.

order………………………………………………………………………………….……..

explanation………………………………………………………………………………

……………………………………………………………………..…………………….

…………………………………………………..……………………………… [1]

(iii) Calculate the gradient of the line, showing your working clearly on the graph.

gradient = ………………………cm^3 min^{-1} [2]

(iv) The y–intercept of your line gives the volume of sodium thiosulfate required to completely react with the iodine present if an aliquot is taken at t_d = 0.0 min.

Read from your graph and record this volume of **FA 5**, $\boldsymbol{V_{max}}$. Use this value to calculate the concentration of iodine in **FA 4**.

volume of **FA 5**, $\boldsymbol{V_{max}}$ = …………………..cm^3

concentration of iodine in **FA 4** = ……………………mol dm^{-3} [3]

(c) Use the information provided on page 6 and your answer to **2(b)(ii)**, write an expression for the rate equation for reaction 1. Include the units for rate in your answer.

Rate = …………………………………..…………………………………………………….. [2]

(d) In step 6 of the experimental procedure, the aliquot is transferred into the second conical flask containing **FA 6**.
Explain why it is necessary to add **FA 6**, and how the titre values will be affected with the omission of **FA 6**.

…………………………………………………………………………………………………

…………………………………………………………………………………………………

………………………………………………………………………………………………… [2]

(e) Suggest how you would carry out a modified experiment of the one that you performed in **2(a)** to verify the order with respect to propanone.

On the axes in Fig. 2.2, sketch the graph you have obtained in **2(b)(i)** and the graph you would expect to obtain from the modified experiment. Label both graphs clearly.

Explain your answer.

Fig. 2.2

Modification…………………………………………………………………………………

…………………………………………………………………………………………………..

explanation ………………………………………………………………………………..

………………………………………………………………………………………………… [3]

[Total: 20]

3 Qualitative Analysis

FA 7 is a solid oxide of an unknown metal cation **Z**. This oxide has an M_r **not** exceeding 140.

[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 8 is solid potassium ethanedioate, $K_2C_2O_4$.

FA 9 is an aqueous organic compound containing one functional group.

You will perform tests to determine:

- the metal cation **Z** in **FA 7**
- the functional group present in **FA 9**

(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
(i) Add all of the solid **FA 8** into a boiling tube. Add 15 cm^3 of **FA 2** into this boiling tube. Using a hot water bath, gently warm the mixture in the boiling tube until all solid dissolves.	
(ii) Add a spatula of **FA 7** to the mixture. Filter the mixture into another test tube.	
(iii) To 1 cm depth of the filtrate in a test tube, add aqueous sodium hydroxide dropwise, until in excess.	

[4]

(b) Write an equation that explains the observation to the chemical test carried out on the gas evolved in test **(a)(ii)**.

………

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

(c) What is the chemical role of **FA 7** in the reaction between **FA 7** and **FA 8**? 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 7** and explain your reasoning by referring to your answer in **(c)** and the information provided on page 12.

………

………

………

…………………………………………………………………………………………………… [2]

(e) You are provided with an organic solution **FA 9** which contains one functional group.

> **Care: FA 9 is flammable. Do not use Bunsen burner for heating.**
> **Use the hot water provided if heating is required.**

FA 9 gives a positive test with 2,4–dinitrophenylhydrazine.

Devise one other confirmatory test using the bench reagents provided to identify the functional group present in **FA 9**.

Carry out the test. Record details of the test performed and observations made in Table 3.2.

Table 3.2

Confirmatory Test	*Observations*

Functional group present in **FA 9**: ……………………………

[3]

[Total: 12]

[BLANK]

4 Planning

The labels for a bottle of carbonic acid and a bottle of citric acid were mixed up. Carbonic acid is dibasic and citric acid is tribasic.

The reaction between the acid and NaOH can be represented by this general equation

$$H_xA + xNaOH \rightarrow Na_xA + xH_2O, \text{ where } x = 2 \text{ or } 3$$

A series of experiments can be performed where increasing volumes of acid and decreasing volumes of NaOH(aq) are mixed and the temperature rise, ΔT, for each experiment is determined.

In each of the experiments using different volumes of H_xA(aq) and NaOH(aq), the total volume has to be kept constant. 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 H_xA(aq) used will give 2 straight lines of best–fit.

The total volume for each experiment should be kept constant at 60.0 cm^3.

You are to plan a series of **six** experiments so that a graph of ΔT against volume of acid can be plotted to determine the basicity of the acid.

You are provided with:

- 1.00 mol dm^{-3} sodium hydroxide, NaOH
- 1.00 mol dm^{-3} acid from one of the bottles, H_xA
- the equipment normally found in a school or college laboratory.

(a) **(i)** Calculate the volumes of acid required for complete reaction if the acid is dibasic and tribasic respectively.

[1]

(ii) Using your answers in **(i)**, use the space provided below to tabulate the volumes of acid and NaOH in each experiment.

[1]

(b) In your plan to determine the basicity of H_xA, you should include brief details of:

- the apparatus you would use;
- the procedure you would follow;
- the measurements you would make. [4]

(c) **(i)** Sketch on Fig. 4.1 you would expect to obtain from your results if the acid is a dibasic acid. Explain your answer.

Fig 4.1

Explanation ………………………………………………………………………………………

……

……

……

……

……

[2]

(ii) Describe how you would make use of the graph to determine the value of enthalpy change of neutralisation, ΔH_{neut} .

[1]

[Total: 9]

[BLANK]

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 → (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 aq. 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