NATIONAL JUNIOR COLLEGE

SH2 Year-End Practical Examination

Higher 2

CANDIDATE NAME			
SUBJECT CLASS		REGISTRATION NUMBER	

CHEMISTRY **9729/04**

Paper 4 Practical **17 August 2017**

Candidates answer on the Question paper **2 hours 30 minutes**

Additional Materials: As listed in the Confidential Instructions

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 19 and 20.

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	/ 12
2	/ 16
3	/ 10
4	/ 17
Total	/ 55

This document consists of **20** printed pages including this cover page.

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

1 Determination of x in the formula of hydrated magnesium carbonate, $MgCO_3.xH_2O$.

Magnesium carbonate is a white, powdery compound that occurs naturally as anhydrous and several hydrated forms. Due to its non-toxicity, it is widely marketed in common products such as cosmetics and toothpaste.

A sample of a hydrated magnesium carbonate mineral is analysed to find out the number of moles of water per mole of magnesium carbonate.

You are given the following:

FA 1 is $MgCO_3.\mathbf{x}H_2O$ solid.

FA 2 is 2.00 mol dm^{-3} nitric acid, HNO_3.

FA 4 is 9.10 g dm^{-3} sodium hydroxide, NaOH.

Indicator: methyl orange

In this question, you will perform a back titration. The data from this titration will be used to determine the *M*r of $MgCO_3.\mathbf{x}H_2O$ and hence the value of **x**.

(a) In this titration, you will react $MgCO_3.\mathbf{x}H_2O$ with an accurately measured amount of excess **FA 2**. The reaction mixture was then used to prepare bulk solution **FA 3**. The amount of unreacted **FA 2** in **FA 3** is then determined *via* titration with **FA 4**. The end-point is reached when the colour of the solution changes from pink to yellow.

(i) Preparation of FA 3 by reacting FA 1 with excess FA 2

Record all the mass measurements in the space provided on page **3**.

1. Weigh out accurately about 0.20 g of **FA 1** in a clean and dry weighing bottle.

2. Transfer **FA 1** to a clean and dry small beaker. Measure and record the mass of weighing bottle and residual solid. Calculate the actual mass of **FA 1** transferred.

3. Pipette 25.0 cm^3 of **FA 2** to dissolve ALL solid **FA 1.**

4. Transfer the resultant solution to a 250 cm^3 volumetric flask.

5. Use a small volume of deionised water to rinse the inner wall of the small beaker and the glass rod. Transfer ALL washings to the volumetric flask.

6. Add more deionised water to make up the volume to the mark. Stopper and shake to ensure complete reaction and to obtain a uniform solution.

7. Label this solution as **FA 3**.

(ii) Titration of FA 3 against FA 4

8. Fill the burette with **FA 4**.

9. Using a pipette, transfer 25.0 cm^3 of **FA 3** into a conical flask and titrate the unreacted HNO_3 with **FA 4**, using methyl orange as indicator.

10. Run **FA 4** from the burette into this flask until solution changes from pink to yellow.

11. Record your titration results in the spaces provided below. Make certain that your recorded results show the precision of your instrument used.

12. Repeat steps **9** to **11** until your results are within ± 0.10 cm^3.

Results

[5]

(iii) From your titrations, obtain a suitable volume of **FA 4** to be used in your calculations. Show clearly how you obtained this volume.

Volume of **FA 4 =** …… [1]

(b) **(i)** Using your titration results, calculate the amount of unreacted HNO_3 in 25.0 cm^3 of **FA 3**. Hence calculate the total amount of unreacted HNO_3 in 250 cm^3 of **FA 3**.
[*A*r: H, 1.0; O, 16.0; Na, 23.0]

Amount of unreacted HNO_3 in 250cm^3 of **FA 3** = ……. [1]

(ii) Determine the total amount of HNO_3 in 25.0 cm^3 of **FA 2** before reacting with $MgCO_3.\mathbf{x}H_2O$.

Amount of HNO_3 in 25.0 cm^3 of **FA 2** = ……...

(iii) Hence calculate the amount of HNO_3 used to react with $MgCO_3.\mathbf{x}H_2O$.

Amount of HNO_3 reacted with $MgCO_3.\mathbf{x}H_2O$ = ……. [1]

(iv) Determine the relative molecular mass of $MgCO_3.\mathbf{x}H_2O$.

M_r of $MgCO_3.\mathbf{x}H_2O$ = …….

Hence calculate the value of **x** in $MgCO_3.\mathbf{x}H_2O$. Give your answer to the nearest whole number.
[*A*r: H, 1.0; C, 12.0; O, 16.0; Mg, 24.3]

x is ……..…….[2]

(c) Another student repeated the experiment using the procedure in **(a)**, however in **step 6**, he made up the solution to the 250 cm^3 mark by adding **FA 2** instead of deionised water. Explain the effect on his titre value and why it is unsuitable to do so.

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……[2]

[Total:12]

2 Determination of the enthalpy change for the formation of hydrated magnesium carbonate, $\Delta H_{reaction1}$.

In this experiment, you are to determine $\Delta H_{reaction3}$.
$\Delta H_{reaction1}$ will be determined using Hess's law.

$Mg(s) + C(s) + (3+\mathbf{x})/2\ O_2(g) + \mathbf{x}H_2(g) \rightarrow MgCO_3.\mathbf{x}H_2O(s)$ $\Delta H_{reaction1}$

$Mg(s) + 2HNO_3(aq) \rightarrow Mg(NO_3)_2(aq) + H_2(g)$ $\Delta H_{reaction\ 2} = -490$ kJ mol^{-1}

$MgCO_3.\mathbf{x}H_2O(s) + 2HNO_3(aq) \rightarrow Mg(NO_3)_2(aq) + CO_2(g) + (\mathbf{x}+1)H_2O(l)$ $\Delta H_{reaction3}$

FA 1 is $MgCO_3.\mathbf{x}H_2O$ solid.

FA 2 is 2.00 mol dm^{-3} nitric acid, HNO_3.

(a) You are to perform the experiment below.

Determination of $\Delta H_{reaction3}$

1. Place a Styrofoam cup with lid inside a second Styrofoam cup which is held in a glass beaker to prevent it from tipping over.
2. Using a measuring cylinder, place 40.0 cm^3 of **FA 2** into the first Styrofoam cup. Measure and record its initial temperature, T_i.
3. Measure accurately about 1.00 g of **FA 1** in a weighing bottle.
4. Transfer **FA 1** into the Styrofoam cup containing **FA 2** and quickly replace the lid. Stir and measure the highest/lowest temperature obtained. Record this temperature, T_f.
5. Reweigh the weighing bottle.
6. Wash and dry the Styrofoam cups.
7. Record your data in the space provided below. Make certain that the recorded results show the precision of the instrument used.

Results

[3]

(i) Using your results, calculate the amount of heat change, $\Delta q_{reaction3}$, by reaction 3:

[c = 4.18 $Jg^{-1}K^{-1}$, density of water = 1.00 g cm^{-3}]

$$MgCO_3.\mathbf{x}H_2O(s) + 2HNO_3(aq) \xrightarrow{\Delta H_{reaction3}} Mg(NO_3)_2(aq) + CO_2(g) + (\mathbf{x}+1)H_2O(l)$$

$\Delta q_{reaction3}$ = ……[1]

(ii) Calculate the $\Delta H_{reaction3}$.

(If you were unable to calculate **x** in **1(b)(iv)**, you may assume that **x** = 3 for the calculation here and in part **(b)**. Note: this is a hypothetical value.)

$\Delta H_{reaction3}$ = ……[1]

(b) Determination of $\Delta H_{reaction1}$

It is given that ΔH_f^{θ} of $CO_2(g)$ and $H_2O(l)$ is −393.5 kJ mol^{-1} and −285.8 kJ mol^{-1} respectively. Using these values together with $\Delta H_{reaction2}$ and $\Delta H_{reaction3}$, construct an energy cycle and calculate a value for $\Delta H_{reaction1}$.

$\Delta H_{reaction1}$ = ……[2]

(c) **(i)** Calculate the percentage error in the measurement of ΔT in the experiment.

percentage error = …….[1]

(ii) Apart from errors associated with the thermometer, suggest one **significant** source of error in the procedure used in this experiment. Suggest an improvement that could be made to reduce this error.

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……..[2]

(d) Planning

In another experiment, you are to plan an experiment to determine the identities of 3 unknown solutions using experimental techniques from thermochemistry.

You are provided with the following:

- solutions of **FB 1**, **FB 2** and **FB 3**, which can be any of the following:
  - 1 mol dm^{-3} aqueous ammonia
  - 1 mol dm^{-3} potassium hydroxide
  - 2 mol dm^{-3} hydrochloric acid
- the apparatus normally found in a school laboratory

In your plan, you should include

- appropriate quantities of solution used,
- the apparatus you would use and the procedure you would follow,
- the measurements you would take,
- an outline of how you would use your results to identify the 3 solutions. (Note: the use of indicator and litmus paper is not allowed)

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[Total: 16]

3 Determination of the order with respect to Fe^{3+} of the reaction between iron(III) and iodide in acidified medium.

Iodide ions are oxidised by iron(III) ions in the presence of acid.

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

The rate of this reaction can be measured by adding thiosulfate ions, $S_2O_3^{2-}$, and some starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.

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

When all the thiosulfate ions have reacted, the iodine which continues to be produced then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the reaction mixture to turn blue-black.

You are to investigate how the rate of reaction is affected by changing the concentration of iron(III) chloride.

The general rate equation can be expressed as rate = $k [Fe^{3+}]^n[I^-]^p$

As the concentration of iodide is present in high concentration, its concentration stays relatively constant during the course of reaction.

Rate equation thus can be rewritten as: rate = $k' [Fe^{3+}]^n$

Taking the logarithm function of the whole equation gives:

$$\lg \text{rate} = \lg k' + n\lg[Fe^{3+}]$$

By plotting a suitable graph, the gradient gives the order of reaction with respect to Fe^{3+}.

You are provided with the following

FA 5 is 0.0500 mol dm^{-3} aqueous sodium thiosulfate, $Na_2S_2O_3$.

FA 6 is 0.500 mol dm^{-3} aqueous potassium iodide, KI.

FA 7 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

FA 8 is 0.0500 mol dm^{-3} aqueous iron(III) chloride, $FeCl_3$.

Starch solution

(a) Procedure

Experiment 1

1. Fill a burette with **FA 5** and another burette with **FA 8**.

2. Run 20.00 cm^3 of **FA 8** from the burette into a 250 cm^3 conical flask.

3. Use a suitable measuring cylinder to add 20 cm^3 of **FA 7** to the flask.

4. Place a magnetic stir bar into the flask and set it stirring at a constant speed of 600 rpm.

5. Using another suitable measuring cylinder, transfer 20 cm^3 of **FA 6** into a 100 cm^3 beaker.

6. Add to the beaker from the burette 1.00 cm^3 of **FA 5**.

7. Add 1 cm^3 of starch indicator to the mixture in the beaker.

8. Tip the contents of the beaker into the conical flask and immediately start a stopwatch.

9. Observe the solution and stop the time when the solution turns blue-black.

10. Record the time taken to the nearest second.

Experiment 2

Repeat step 1 – 10 **except in step 2**, run 10.00 cm^3 of **FA 8** into the flask and using a suitable measuring cylinder to transfer 10 cm^3 deionised water into the flask.

Experiment 3 – 5

Carry out three further experiments to investigate how the reaction time changes with different volumes of iron(III) chloride.

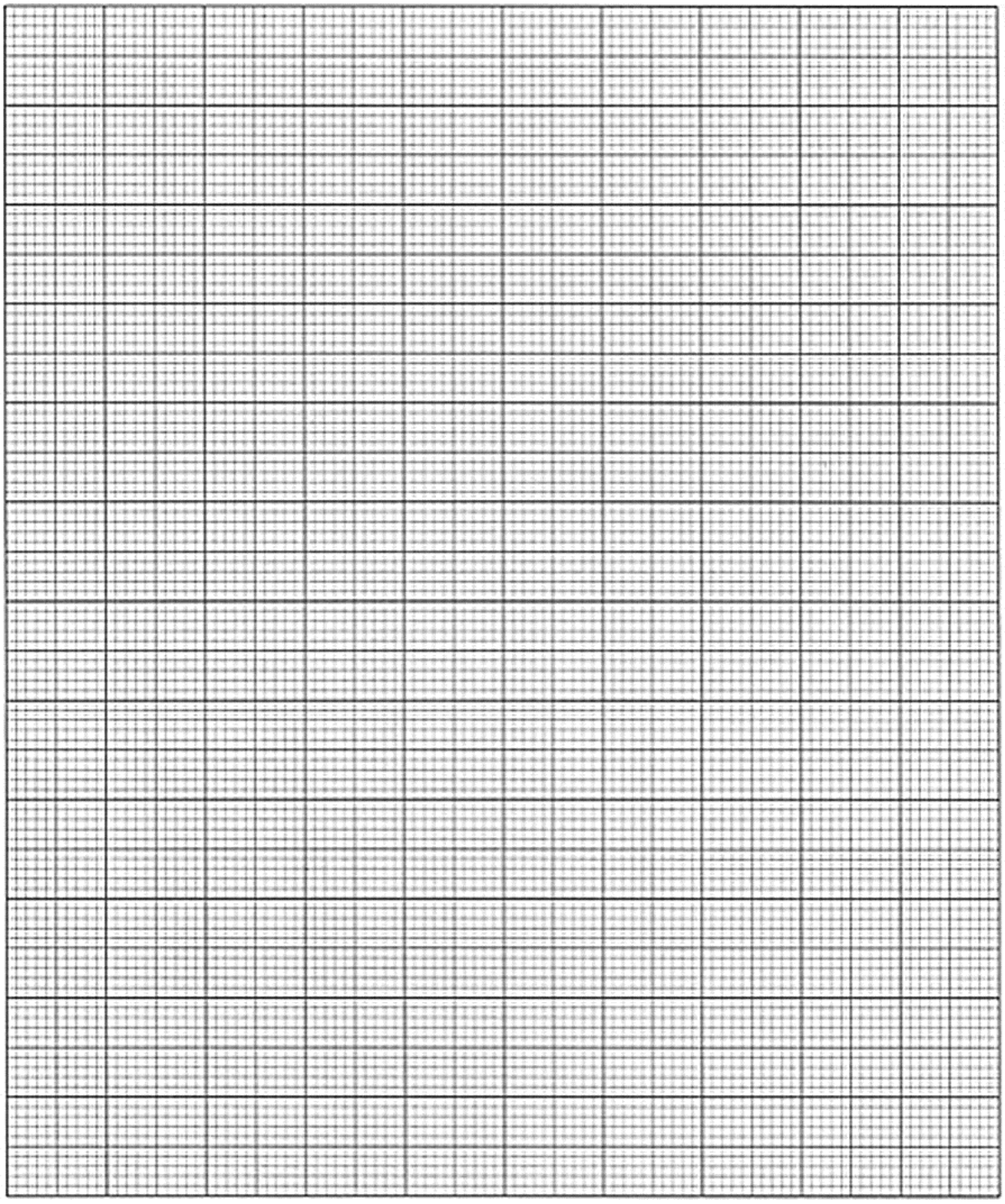
Do not use a volume of **FA 8** that is less than 6.00 cm^3.

Record all your results in a single table. You should include the volume of **FA 8**, the volume of deionised water and the reaction time. Your recorded results should also include calculated values to enable you to plot

lg (1/time) against **lg (volume of FA 8)**

[5]

(b) (i) Use the grid below to plot a graph of **lg (1/time)** against **lg (volume of FA 8)**.



[3]

(ii) From your results, deduce the order of reaction with respect to Fe^{3+}.

order with respect to Fe^{3+} = ……[2]

[Total: 10]

4 Qualitative analysis

In this question, you will deduce the two anions in solution **X** and a cation and an anion in solution **Y**. You will also work with an organic compound **Z**.

You will perform a series of test-tube reactions and use the observations to help you identify the unknown.

Test	Procedure	Observations
(a)	To 6 cm^3 of **X**, add barium nitrate solution until in excess.	
(b)	Filter the mixture from **(a).** Wash and retain the residue for test **(c)**. Collect the filtrate for test **(d)** and **(e)**.	
(c)	To separate portions of the residue **(i)** add 2 cm^3 of hydrochloric acid	
	(ii) add 2 cm^3 of organic compound **Z** [You are to test for any gas evolved]	
(d)	To 1 cm depth of the filtrate from **(b)** in a test-tube **(i)** Add a few drops of organic compound **Z** and warm in a water bath **(ii)** followed by 1 cm^3 of nitric acid and 5 drops of silver nitrate. Add excess ammonia solution.	

(e)	To separate 1 cm depth of **Y** **(i)** add filtrate from **(b)** dropwise until in excess	
	(ii) add NaOH(aq), followed by one spatula of zinc powder and warm (CARE!)	

[5]

(f) **(i)** From the observation in part **(c)(i)** and **(e)(i)**, identify the anion in the residue and the filtrate.

……..[2]

(ii) Suggest two functional groups present in organic compound **Z**. Justify your answer by quoting relevant evidence from the tests carried out.

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……..[2]

(g) Suggest 1 possible identity for the cation and anion present in solution **Y**.

cation: …….. [1]

anion: …….. [1]

(h) Planning

Consider the following organic compounds.

benzaldehyde 2-methylpropan-2-ol butan-1-ol propanal

Plan an investigation, using test-tube reactions, which would allow you to identify each of these four organic compounds.

Each compound should be identified by at least one positive test results. It is **not** sufficient to identify a compound simply by eliminating all the others.

Your plan should include:

- details of the reagents and conditions to be used,
- an outline of the sequence of steps you would follow,
- an explanation of how you would analyse your results in order to identify each compound.

Once a compound has been clearly identified, your plan should concentrate on distinguishing the remaining compounds.

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[6]

[Total: 17]

GO TO NEXT PAGE FOR QUALITATIVE ANALYSIS NOTES

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

anions	***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 by 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 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 potassium manganite(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