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

CANDIDATE NAME			
SUBJECT CLASS		REGISTRATION NUMBER	

CHEMISTRY 9729/04

Paper 4 Practical **Wednesday 19 August 2020**

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 19 and 20.

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

Shift
Laboratory

For Examiner's use	
1	/ 16
2	/ 13
3	/ 13
4	/ 12
Presentation	/ 1
Total	/ 55

This paper consists of **18** printed pages including this cover page.

1 Determination of a value for the number of water of crystallization, *x*, in an iron(II) salt, $(NH_4)_2Fe(SO_4)_2.\ x\ H_2O$.

The iron (II) salt, **$(NH_4)_2Fe(SO_4)_2.\ x\ H_2O$**, can be titrated against potassium manganate (VII) solution. The two half–equations for the reaction are shown below.

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O$$

$$Fe^{2+} \longrightarrow Fe^{3+} + e^-$$

FA 1 is potassium manganate(VII), $KMnO_4$ solution, of concentration **around 0.02 mol dm^{-3}**
FA 2 is 0.0780 mol dm^{-3} ethanedioate ions $C_2O_4^{2-}$
FA 3 is a solution of the iron(II) salt containing 38.4 g dm^{-3} of $(NH_4)_2Fe(SO_4)_2.\ x\ H_2O$
FA 4 is dilute sulfuric acid, H_2SO_4

The exact concentration of **FA 1** can be determined when **FA 2** is titrated against it.

$C_2O_4^{2-}$ reacts with MnO_4^- in an acidic medium to form Mn^{2+} and CO_2 gas.

$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \longrightarrow 2Mn^{2+} + 8H_2O + 10CO_2$$

As this reaction is very slow at the start, the $C_2O_4^{2-}$ solution is heated prior to titration against MnO_4^-. However, it is not necessary to continuously heat the solution as the rate of reaction will increase as the reaction proceeds.

Method

(a) (i) Standardisation of FA 1 solution

1. Fill the burette with **FA 1**.
2. Using a pipette, transfer 10.0 cm^3 of **FA 2** into the conical flask.
3. Using an appropriate measuring cylinder, transfer 20 cm^3 of **FA 4** to the same conical flask.
4. Place a 1 ^{o}C thermometer into this conical flask.
5. Heat the contents of the conical flask using the bunsen burner.
6. **Turn off the Bunsen burner** once a temperature of 60 ^{o}C is reached.

Caution: The conical flask should be comfortable to the touch. However, you may also use the paper towels to handle the neck of the heated flask if it is too hot for you.

7. Using a small volume of deionised water, wash down the sides of the thermometer and remove it from the conical flask.
8. Add **FA 1** from the burette into this flask until a **permanent** pale pink colour is obtained. Initially, the colour of FA 1 will take some time to disappear, continue swirling. Subsequently, as the reaction proceeds, the rate of reaction will increase.
9. Record your titration results in the space provided on page **3**.

10. Repeat steps **1** to **7** as necessary until consistent results are obtained.

Results:

[3]

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

Volume of **FA 1** used = …….

[1]

(iii) Hence, determine the concentration of MnO_4^- in **FA 1**.

Concentration of MnO_4^- in **FA 1** = …….

[2]

(iv) For the titration in **(a)(i)**, the $C_2O_4^{2-}$ solution needs to be at about 60 °C at the start.

During the titration, as **FA 1** is added, the temperature of the mixture decreases. However, this decrease in temperature did not cause the rate of reaction between $C_2O_4^{2-}$ and MnO_4^- to decrease.

(i) Suggest a reason for the slow reaction at the start.

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[1]

(ii) Explain why the rate of reaction did not decrease even though the temperature of the mixture decreased.

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[1]

(v) Calculate the maximum total percentage uncertainty for your titration in **(a)(i)** if the uncertainty associated with each reading using a 10.0 cm^3 pipette and a burette are ±0.03 cm^3 and ±0.05 cm^3 respectively.

Percentage uncertainty = ……...…….

[1]

(b) (i) Titration of FA3 against FA1

1. Fill the burette with **FA 1**.
2. Pipette 25.0 cm^3 of **FA3** into a conical flask and add 20 cm^3 of **FA 4** using an appropriate measuring cylinder.
3. Titrate **FA 3** with **FA 1** until an orange-pink colour is obtained.
4. Carry out as many titrations as you think necessary to obtain consistent results. Record your titration results in the space on the next page.

Results:

[2]

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

Volume of **FA 1** used = …….

[1]

(c) Calculations

(i) Calculate the amount of iron(II) ions present in 1.00 dm^3 of **FA 3**.

Amount of iron(II) ions in 1.00 dm^3 of **FA 3** = …….

[2]

(ii) Using your answer in **(c)(i)**, determine the value of x, the number of water of crystallisation in the iron(II) salt, $(NH_4)_2Fe(SO_4)_2 . x\ H_2O$.

[A_r: Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

x = ...

[2]

[Total: 16]

2 Determination of a value for the enthalpy change, ΔH, of the reaction between magnesium and hydrochloric acid.

Magnesium reacts with an excess of hydrochloric acid according to the following equation.

$$Mg(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2(g)$$

FA 5 is 2 mol dm^{-3} hydrochloric acid, HC*l*

FA 6 is magnesium ribbon, Mg

You are also provided with sandpaper

(a) Method

1. Place a Styrofoam cup in a 250 cm^3 beaker to prevent it from tipping over.

2. Sand a piece of **FA6**, weigh and record its mass in the space below.

3. Using a measuring cylinder, transfer 25 cm^3 of **FA 5** into the Styrofoam cup.

4. Measure the temperature of **FA 5** in the Styrofoam cup using the 0.2 °C thermometer. This is the temperature at time = 0 minute.

5. Start the stopwatch and leave it running for the whole experiment.

6. Record the temperature of the acid every half a minute for 2 minutes.

7. At time = 2.5 minutes, carefully drop the piece of **FA 6** into the acid and stir the mixture. Do not measure the temperature.

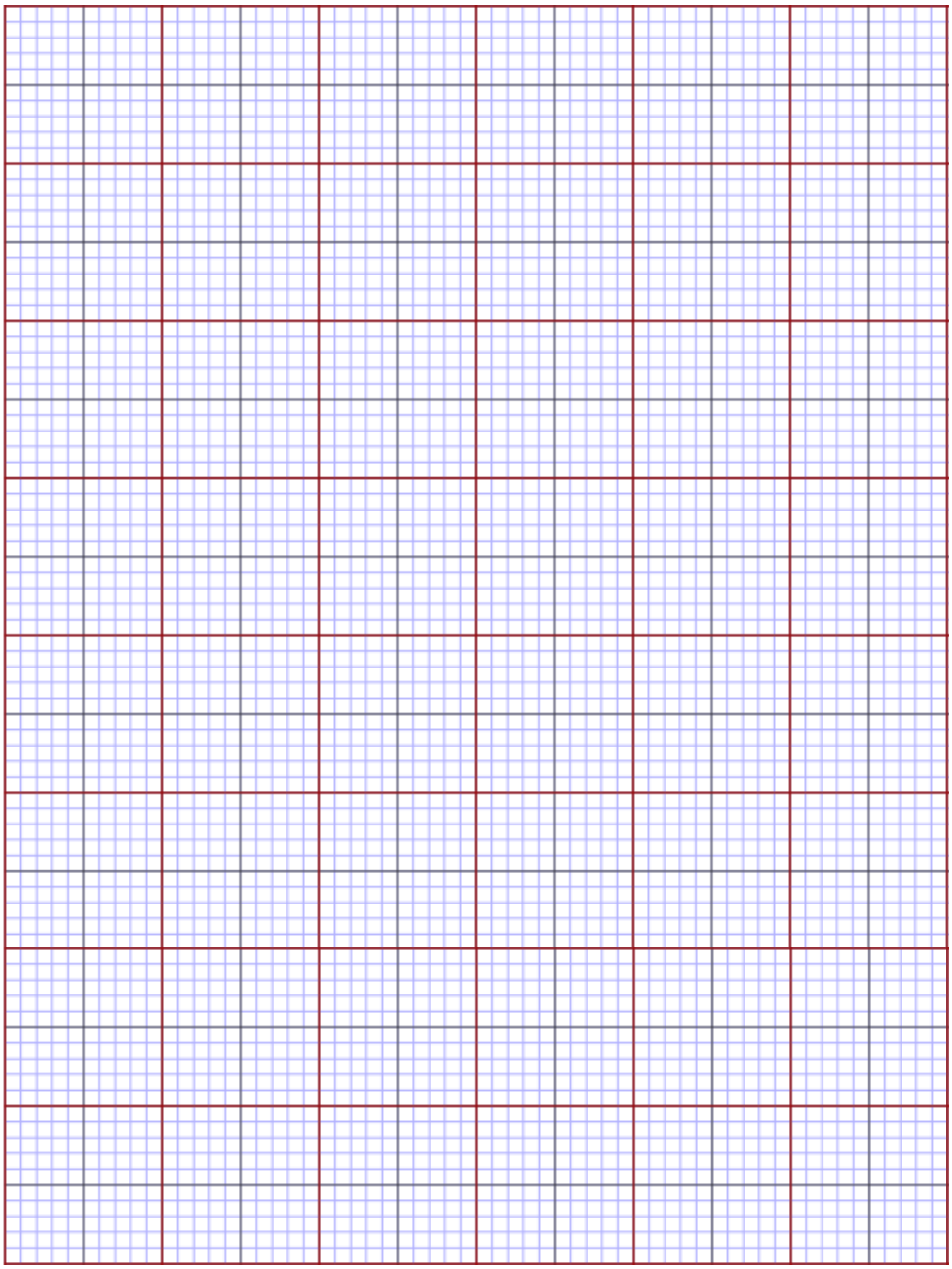
8. Record the temperature of the mixture at time = 3 minutes, then every half a minute over a total duration of 8 minutes. Stir the mixture between thermometer readings.

Results:

Mass of **FA6** /g	

[4]

(b) Plot a graph of temperature against time on the grid below.



[3]

Draw two lines of best fit through the points on your graph, the first for the temperature before adding **FA 6** and the second for the cooling of the mixture once the reaction is complete.

Extrapolate the two lines to time = 2.5 minutes, draw a vertical line between the two and determine the theoretical rise in temperature at this time.

Theoretical rise in temperature at 2.5 minutes = ... °C

[1]

(c) Calculation

(i) Use your answer to **(b)** to calculate the heat energy, in joules, given out when **FA 6** is added to **FA 5**.

(Assume 4.2 J of heat energy raises the temperature of 1.0 cm^3 of the mixture by 1.0 °C)

Heat energy evolved = ……. J

[1]

(ii) Use your answer to **(i)** to calculate the enthalpy change, in kJ mol^{-1}, when 1 mole of **FA 6** reacts with **FA 5**.

[A_r: Mg, 24.3; H, 1.0; C*l*, 35.5]

Enthalpy change = ……. kJ mol^{-1}

[2]

(d) A student carried out the same procedure using the same concentration of sulfuric acid, H_2SO_4, instead of hydrochloric acid and predicted that the enthalpy change would be twice of that with hydrochloric acid. Comment on this prediction.

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[1]

(e) The enthalpy change determined in **(ii)** is only an approximation of the actual value. Suggest and explain one improvement you would make to the method in **(a)** to increase the accuracy of the experiment.

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[1]

[Total: 13]

3 Investigation of some inorganic reactions.

You are to perform the tests described in **Table 3.1** and record your observations in the table.

In all tests, the reagents should be added gradually until no further change is observed unless you are instructed otherwise. You should indicate clearly at which stage in a test a change occurs, recording your observations alongside the relevant tests.

The volumes given below are approximate and should be estimated rather than measured, unless otherwise stated. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

No additional or confirmatory tests for ions present should be attempted.

FA 7 and **FA 8** are solutions each containing one cation and one anion.

Use a 1 cm depth of **FA 7** or **FA 8** in a test-tube to carry out the following tests on the two solutions and record your observations.

Table 3.1

	Test	*Observations*	
		FA 7	**FA 8**
(a)	Add aqueous sodium hydroxide.		
(b)	Add aqueous ammonia.		
(c)	**You do not need to carry out these tests.** Add 1 cm depth of dilute hydrochloric acid, then transfer the mixture into a boiling tube and warm gently.	**Brown gas evolved which turns damp blue litmus paper red.**	**No observable change.**
(d)	Add two or three **drops** of **acidified** aqueous potassium manganate (VII). (The potassium manganate (VII) provided is not acidified.)		

	Test	*Observations*	
		FA 7	**FA 8**
(e)	Add 1 cm depth of aqueous barium nitrate, then add dilute hydrochloric acid.		

Identify as many of the ions present in **FA 7** and **FA 8** as possible from your observations. If you are unable to identify any of the ions from your observations, write 'unknown' in the space.

	FA 7	**FA 8**
Cation		
Anion		

[8]

(f) **FA 9** is a solid with an anion containing the same element as one of the anions in either **FA 7** or **FA 8** but in a different oxidation state. Relevant anions are listed in the Qualitative Analysis Notes on page 18.

Place a spatula of **FA 9** in a test tube and add 2 cm depth of deionised water. Shake to dissolve the solid and make a solution of **FA 9**.

(i) Select reagents to test whether the anion in **FA 9** contains the same element as the anion in **FA 7**, or in **FA 8**.

Carry out your test(s) on the solution of **FA 9** and record your observations and conclusions below.

	Test	*Observations*	*Conclusion*
(f)			
(g)			

[5]

[Total : 13]

4 Planning (P)

FB 1 is a Group 2 metal chloride hydrate, MCl_2. n H_2O. In order to determine the identity of M and the value of n, two different methods – volumetric and gravimetric analysis were carried out. The volumetric analysis depends on an application of the principles of solubility product, while gravimetric analysis is used to measure the mass loss on forming the anhydrous metal chloride.

(a) Volumetric analysis

The molar mass of the metal chloride hydrate, MCl_2. n H_2O can be determined by titrating a solution of **FB 1** with aqueous silver nitrate. The equation for this reaction is shown below.

$$Ag^+(aq) + Cl^-(aq) \longrightarrow AgCl(s)$$

Data for use in this question are given in **Table 1**.

Table 1

	colour	**K_{sp} (at 25°C)**
$AgCl$	white	2.02×10^{-10} mol^2 dm^{-6}
Ag_2CrO_4	red	3.01×10^{-12} mol^3 dm^{-12}

FB 1 is the Group 2 metal chloride hydrate, MCl_2. n H_2O(s)

FB 2 is 0.200 mol dm^{-3} silver nitrate

FB 3 is neutral chromate indicator, K_2CrO_4(aq)

The following procedure was carried out.

Step 1 Weigh 6.002 g of **FB 1** using a weighing bottle and transfer the contents completely into a 100 cm^3 beaker.

Step 2 Add deionised water to the beaker and stir until the solid has dissolved.

Step 3 Transfer the contents and washings carefully into a 250 cm^3 volumetric flask and top up the volumetric flask with deionised water to the 250 cm^3 mark. This solution is labelled as **FB 4**.

Step 4 Use a pipette to transfer 25.0 cm^3 of **FB 4** into a conical flask.

Step 5 Titrate the solution with **FB 2** using 10 drops of **FB 3** indicator.

(i) Aqueous silver nitrate is separately added to solutions containing 0.1 mol dm^{-3} $Cl^-(aq)$ and 0.01 mol dm^{-3} $CrO_4^{2-}(aq)$.

Calculate the concentration of Ag^+ that must be present to cause the precipitation of

- AgC*l*,

- Ag_2CrO_4.

[2]

(ii) Using the data given in **Table 1** and your answer to **(a)(i)**, suggest what you would see at the beginning of the titration and at the end–point, and explain why $K_2CrO_4(aq)$ can be used as an indicator in this titration.

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[2]

(iii) **FB 3** contains a neutral solution of $K_2CrO_4(aq)$. Suggest a reason why the titration cannot be carried out in an alkaline condition.

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[1]

(iv) In the experiment, 24.60 cm^3 of **FB 2** was used to completely react with 25.0 cm^3 of **FB 4**.

Outline how this result can be used to determine the molar mass of MCl_2. n H_2O.

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[3]

(b) Gravimetric analysis

Write a plan to measure the mass loss on forming the anhydrous metal chloride salt, so that the identity of *M* and the value of *n* can be determined.

You may assume that you are provided with the following.

- 5 g of solid **FB 1**
- the apparatus normally found in a school or college laboratory

Your plan should include:

- details of the experimental procedure;
- details to ensure reliability of the results;
- how your results would be recorded and presented in a table form;
- how you would use your results and answer to **(a)(iv)** to determine the value of *n* and the identity of *M*.

You may find it useful to use the letters *a*, *b*, *c* etc. to represent each **measurement** of mass.

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[4]

[Total: 12]

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 Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al 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