		UNIOR COLLEG	
CANDIDATE NAME			
SUBJECT CLASS		REGISTRATION NUMBER	
CHEMISTRY			9729/04
Paper 4 Practical			Tuesday 17 August 2021
Candidate answer on the	Question paper.		2 hours 30 minutes
READ THESE INSTRUC	TIONS FIRST		
Write your identification n Give details of the practic Write in blue or black pen	al shift and laborato		, in the boxes provided.
You may use an HB pend Do not use staples, paper	oll for any diagrams oclips, glue or corre	or graphs. ction fluid.	Shift
Answer all questions in Paper.	the spaces provid	ded on the Question	
тарст.			Laboratory
The use of an approved appropriate. You may lose or if you do not use appro	e marks if you do n	r is expected, where ot show your working	

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.

For Examiner's use		
1	/ 15	
2	/ 15	
3	/ 14	
4	/ 10	
Presentation	/1	
Total	/ 55	

This document consists of 19 printed pages and 1 blank page.

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Determination of the concentration of a solution of sodium hydroxide and the enthalpy change of neutralisation, ΔH_n

FA 1 is 1.0 mol dm⁻³ sulfuric acid, H₂SO₄.

FA 2 is a solution of sodium hydroxide, NaOH, of unknown concentration.

According to the *Arrhenius* theory of acids and bases, an acid produces H⁺(aq) ions and a base produces OH⁻(aq) ions, in aqueous solution. Using the *Arrhenius* theory, an acid-base neutralisation reaction involves reacting these two ions together to produce water molecules. The equation for this neutralisation reaction is given below.

$$H^{+}(aq) + OH^{-}(aq) \rightarrow H_{2}O(l)$$

 $\Delta H_{\rm n}$

In this question, you will carry out a series of experiments where different volumes of **FA 1** and **FA 2** are mixed together.

You will determine the temperature change of the mixture, ΔT of each experiment and then analyse your results graphically in order to determine the

- concentration of NaOH in FA 2
- maximum temperature change, ΔT_{max}
- value for the enthalpy change of neutralisation, ΔH_n

(a) Method

- Support the Styrofoam cup in a 250 cm³ beaker.
- Using a measuring cylinder, place 20.0 cm³ of **FA 2** into the Styrofoam cup.
- Measure the temperature of **FA 2** in the Styrofoam cup. Record the initial temperature of the solution of **FA 2** as **T**_{initial}.
- 4 Place 40.0 cm³ of FA 1 into another measuring cylinder.
- Transfer the **FA 1** in the measuring cylinder into the Styrofoam cup, stir and record the maximum temperature obtained in the reaction as T_{max} .
- 6 Rinse and dry the Styrofoam cup and the thermometer.
- Repeat steps 2 to 6 using volumes of FA 2 listed in Table 1.1. Fill in Table 1.1 with the appropriate volumes of FA 1 to be used in each experiment such that the total volume of the reaction mixture is 60.0 cm³.

Table 1.1

Experiment	1	2	3	T 4		
Volume of FA 2 / cm ³	20.0	30.0	40.0	50.0	5	6
Volume of FA 1 / cm ³	40.0					

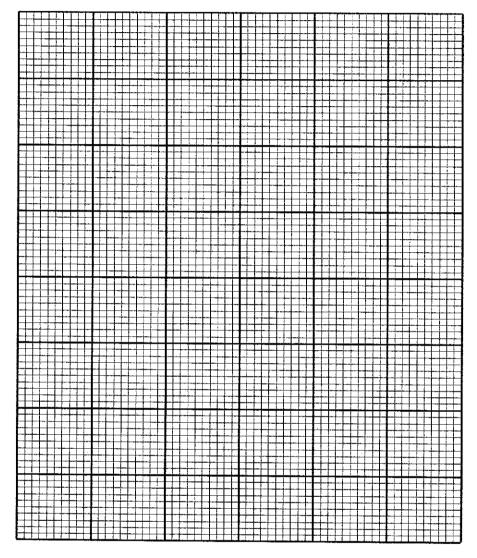
[1]

In an appropriate format in the space provided, prepare a table in which to record for each experiment

- the initial temperature, T_{initial}
- the maximum temperature, T_{max}
- the change in temperature, ΔT

(b) (i) On the grid given below, plot a graph of ΔT (y-axis) against volume of **FA 2** used (x-axis) using the data you have obtained in (a).

By considering the points you have plotted, carry out two more experiments (experiment 5 and 6) which will enable you to identify the volume of FA 2 that gives the maximum temperature change, ΔT_{max} .



[2]

Given that $\Delta T = 0$ at volume of FA 2 = 0.0 and 60.0 cm³, draw two straight lines of best fit. The first best fit line should be drawn using the plotted points where ΔT is increasing and the second best fit line should be drawn using the plotted points where ΔT is decreasing. Extrapolate these two lines until they cross.

(iii) From your graph, determine the maximum temperature change, ΔT_{max} , and the volume of FA 2 required to obtain this.

 $V_{FA2} = cm^3$

[1]

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	16.1		22 T I	α	•

(c) Using your answers in b(iii),

	(i)	calculate the concentration of NaOH in FA 2
		concentration of NaOH in FA 2 =
		[2]
	/::\	
	(ii)	calculate the heat evolved at ΔT_{max} .
		You may assume that the specific heat capacity of the reaction mixture is $4.18 \mathrm{J} \mathrm{g}^{-1} \mathrm{K}^{-1}$ and that its density is $1.00 \mathrm{g} \mathrm{cm}^{-3}$.
		heat evolved =
		[1]
. B		
(d)	Using	your answer in (c)(ii), calculate the enthalpy change of neutralisation, ΔH_n .
		$\Delta H_n = \dots$
		[1]

Analysis and Evaluation

(e)	State and explain the effect on the magnitude of ΔT_{max} if ethanedioic acid of the same concentration was used instead of sulfuric acid in the experiment in (a).
	Effect on the magnitude of Δ 7 _{max} :
	explanation:
	·
	[2]
f)	Student A carefully performed the same experiment and correctly processed the data using the same method. His calculated value of ΔH_n is -65.0 kJ mol ⁻¹ while the published value for this enthalpy change is -57.7 kJ mol ⁻¹ .
	The specific heat capacity of the Styrofoam cup has not been taken into consideration in calculating $\Delta \textit{H}_n$.
	Explain whether this omission could have been the reason for the discrepancy between the value obtained by Student A and the one published.
	[2]
	[Total: 15]

2 To determine the concentration of a solution of copper(II) sulfate.

FA 3 is aqueous copper(II) sulfate, CuSO₄(aq), of unknown concentration.

FA 4 is 0.100 mol dm $^{-3}$ sodium thiosulfate, Na₂S₂O₃.

FA 5 is a 10% solution of potassium iodide, KI.

You are also provided with a starch indicator.

When an excess of potassium iodide is added to copper(II) sulfate, iodine and copper(I) iodide are formed. All the copper is precipitated as copper(I) iodide and the iodine turns the solution brown.

$$2CuSO_4 + 4KI \rightarrow 2CuI + 2K_2SO_4 + I_2$$

The iodine produced in this reaction may be titrated against a standard solution of sodium thiosulfate. The equation for this reaction is shown below.

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

When **FA 5** is added to **FA 3**, the solution turns brown due to the formation of iodine. As **FA 4** is added to this mixture, this brown colour fades. If starch indicator is added when the colour is pale brown, the solution immediately turns blue-black as the starch reacts with the residual iodine.

The end-point of this titration is reached when the blue-black colour **just** disappears. The off-white precipitate of copper(I) iodide does not interfere with the reactions involved in this titration. This precipitate may be a light beige at the end-point.

Estimating the required dilution factor.

(a) The solution FA 3 is too concentrated to titrate directly with the FA 4 solution.

You are to perform a preliminary experiment in order to estimate a dilution factor for the FA 3 solution, so that 25 cm³ of this diluted solution would require approximately 25 cm³ of FA 4 for complete reaction. You will do this by counting the number of drops of FA 4 needed to react completely with 5 drops of FA 3.

- 1 Using a dropping pipette, add 5 drops of **FA 3** to a test-tube.
- To this test-tube, add about 1 cm³ of FA 5. You should estimate, not measure, this volume.
- Use another dropping pipette to add FA 4, drop by drop, until the solution is pale brown. Add 5 drops of starch solution and further drops of FA 4 until the blue-black colour just disappears.

The total number of drops of FA 4 you added =

Use your results from this experiment to calculate the volume of FA 3 you would use to make 250 cm³ of the diluted copper(II) sulfate solution, FA 6.

Dilution of FA 3

(b) You will now follow the instructions given below to prepare a diluted solution of FA 3. You will titrate this diluted solution, FA 6, with the FA 4 solution.

Using a burette, measure between 42.50 cm³ and 43.00 cm³ of **FA 3** into the 250 cm³ graduated flask labelled **FA 6**.

Record your burette readings and the volume of FA 3 added to the flask in the space below.

[1]

Make up the contents of the flask to the 250 cm³ mark with deionised water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

- 1 Fill a second burette with FA 4.
- 2 Pipette 25.0 cm³ of **FA 6** into a conical flask.
- 3 Use the measuring cylinder to add 10.0 cm³ of **FA 5** to the flask. An off-white precipitate forms in a brown solution.
- 4 Run **FA 4** from the burette into the flask until the brown colour of the iodine becomes pale brown.
- Add about 10 drops of starch indicator to the flask and continue adding **FA 4** until the blue-black colour **just** disappears, leaving behind the off-white precipitate.
- Record your titration results in the space provided below. Make certain that your recorded results show the precision of your working.
- 7 Repeat points 1 to 6 as necessary until consistent results are obtained.

(c)	From your titrations, obtain a suitable volume of FA 4 to be used in your calculations. Show clearly how you obtained this volume.
	[1]
Cal	culations
(d)	Calculate the number of moles of FA 4 in the volume of FA 4 recorded in (c), and hence deduce the concentration of Cu^{2+} ions, $[Cu^{2+}]$, in FA 6 .
	[Cu²+] in FA € =
	[2]
(e)	© ∂culate the concentration of CuSO₄ in FA 3 .
	the concentration of CuSO ₄ in FA 3 =
	Hence, calculate the mass of $CuSO_4 \cdot 5H_2O$ crystals required to prepare a 1 dm ³ solution of FA 3.
	[A _r : Cu, 63.5; H, 1.0; O, 16.0; S, 32.1]
	the mass of CuSO ₄ •5H ₂ O required =
	[2]

Analy	vsis	and	Eval	uation

(f)	A teacher performs this same experiment, using the quantities described earlier, and obtains a mean titre volume of 24.85 cm ³ .
	The errors (uncertainties) associated with each reading using a graduated flask, pipette and burette are, ±0.15 cm³, ±0.1 cm³ and ±0.05 cm³ respectively.
	Calculate the maximum total percentage error (uncertainty) of this mean titre volume.
	the maximum total percentage error (uncertainty) is%
	[1]
(g)	A second student performs this experiment, using the quantities described earlier. Unfortunately, she was provided with a 9% solution of FA 5 (KI concentration = 90 g dm ⁻³) instead of the 10% KI solution you used (KI concentration = 100 g dm ⁻³).
	Predict, using calculations, whether her titre volumes will be affected.
	[A _r : K, 39.1; I, 126.9]
	[2]
(h)	•
. ,	Identify two different chemical processes that use iodide ions in this experiment.
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	[1]
	[Total: 15]
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3 Investigation of some inorganic reactions.

You are provided with solutions FA 7, FA 8 and FA 9.

You are to perform the tests described in **Tables 3.1** to **3.4**, and record your observations in the tables.

At each stage of any test, you are to record details of the following:

- colour changes seen
- the formation of any precipitate and its solubility in an excess of the reagent added
- the formation of any gas and its identification by a suitable test
- if there is no observable change, write no observable change

You should indicate clearly at which stage in a test a change occurs, recording your observations alongside the relevant tests.

In all tests, the reagents should be added gradually until no further change is observed unless you are instructed otherwise. If any solution is warmed, a **boiling tube** must be used. Rinse and reuse test-tubes where possible.

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

FA 7, FA 8 and FA 9 are salt solutions.

Each salt solution contains one cation and one anion, all of which are different.

- FA 7 contains a halide ion.
- FA 8 and FA 9 contains either a sulfur-containing anion or a nitrogen-containing anion.
- FA 9 contains Na⁺ cation.

(a)

Table 3.1

	Test	Observations
(i)	Add 1 cm depth of FA 7 to a test-tube. Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.	
	Transfer the mixture into a boiling tube and carefully warm the boiling tube and its contents. Do not heat until the mixture boils.	
(ii)	Add 1 cm depth of FA 8 to a test-tube. Add aqueous ammonia slowly, with shaking, until no further change is seen.	

(iii)	Add 1 cm depth of FA 8 to a test-tube.	
	Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.	
	Change is seen.	

Identify the cation present in FA 7 and FA 8 from your observations in Table 3.1. If the tests you carried out did not allow you to identify the cation, write 'unknown' in the space.

	FA 7	FA 8
Cation		

[4]

(b)

Table 3.2

Test	Obser	vations
	FA 8	FA 9
To separate test-tubes each containing 1 cm depth of FA 8 and FA 9,		
add 1 cm depth of dilute sulfuric acid, followed by one drop of aqueous potassium manganate(VII).		

Using the information below about redox properties and your observations in **Table 3.2**, identify the possible sulfur-containing anion and nitrogen-containing anion present in **FA 8** or **FA 9**.

anion	property
nitrite	easily oxidised
nitrate	cannot be oxidised
sulfite	easily oxidised
sulfate	cannot be easily oxidised

Possible anions present in FA 8:	
Possible anions present in FA 9:	[3]

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(c)

Table 3.3

Test	Observations		
	FA 8	FA 9	
To separate boiling tubes each containing 1 cm depth of FA 8 and FA 9 ,			
add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.			
warm the mixture gently and carefully then			
add a piece of aluminium foil to the mixture.			

Using your deductions in (b) and observations in Table 3.3, identify the anion present in FA 8 and FA 9.

	FA 8	FA 9
Anion		

[3]

(d) From the bench reagents provided, suggest reagents you would use to identify the halide ion present in FA 7. Carry out the test and record your observations in Table 3.4.

reagents used:

Table 3.4

Test	Observations

Halide ion present in FA 7:

[3]

(e) The following tests were carried out on another solution, **solution P**. Using the observations given in **Table 3.4** as well as the Qualitative Analysis Notes on pages 19 -20, deduce the identity of **solution P**.

[solution P contains a cation listed in the Qualitative Analysis Notes]

Table 3.4

	Test	Observations
	rest	solution P
(i)	Add aqueous ammonia to 1 cm depth of solution P , until in excess.	No observable change.
(ii)	Add aqueous sodium hydroxide to 1 cm depth of solution P, until in excess.	No observable change.
	If no precipitate forms, transfer the mixture into a boiling tube and carefully warm the boiling tube and its contents. Do not heat until the mixture boils.	No gas evolved.
(iii)	In a separate test-tube, add 1 cm depth of dilute hydrochloric acid to 1 cm depth of aqueous sodium carbonate. Bubble the gas evolved into 1 cm depth of solution P.	Colourless odourless gas evolved, formed white precipitate in solution P .

Identity of solution P:	***************************************	•	[1]
			. .

[Total: 14]

4	- DI	
4	Pla	anning

Many hydrated salts lose water of crystallisation when heated. In this question, you are to plan a procedure that would provide sufficient data to allow you to identify the metal in a hydrated Group 13 sulfate salt, X•16H₂O by heating the salt until it becomes anhydrous.

The equation for the dehydration of the hydrated Group 13 sulfate salt, X•16H₂O, is as shown.

$$X \cdot 16H_2O(s) \rightarrow X(s) + 16H_2O(g)$$

FB 1 is the hydrated salt, X•16H₂O.

(a) Plan an investigation to identify the metal in a hydrated Group 13 sulfate salt, X•16H₂O by heating the salt until it becomes anhydrous.

You may assume that you are provided with:

- 2.0 g of **FB 1**,
- crucible and its lid,
- equipment normally found in a school or college laboratory.

In your plan, you should include brief details of:

- the apparatus you would use,
- the procedure you would follow,
- a table of measurements you would make, using alphabets A, B, C, D etc to represent the measurements made

[5]

how you would ensure that the salt becomes anhydrous

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(b)	Explain how you would use the mass of anhydrous salt to calculate the number of moles of anhydrous residue and hence the relative formula mass of X.
	[2]
(c)	Using a value of 352.5 as the relative formula mass of X, a Group 13 sulfate salt, calculate the relative atomic mass of the metal and identify the metal in X.
	[A;: B, 10.8; A/, 27.0; Ga, 69.7; In, 114.8; T/, 204.4]
	[2]
(d)	Suggest why using a crucible is better than a boiling tube in this experiment.
	[1]
	[Total: 10]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	reaction with >		
calluir	NaOH(aq)	NH3(aq)	
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH₄⁺ (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), Cr³⁺(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, Zn²+(aq)	white ppt. soluble in excess	white ppt. soluble in excess	

(b) Reactions of anions

anion	Reaction	
carbonate,	CO₂ liberated by dilute acids	
chloride, C <i>I</i> ⁻ (aq)	gives white ppt. with Ag+(aq) (soluble in NH₃(aq))	
bromide, Br (aq)	gives pale cream ppt. with Ag+(aq) (partially soluble in NH₃(aq))	
iodide, I ⁻ (aq)	gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq))	
nitrate, NO ₃ -(aq)	NH₃ liberated on heating with OH⁻(aq) and A/ foil	
nitrite, NO ₂ -(aq)	NH₃ liberated on heating with OH⁻(aq) and A/ foil; NO liberated by dilute acids (colourless NO → (pale) brown NO₂ 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, Cl2	greenish yellow gas	pale yellow	pale yellow
bromine, Br2	reddish brown gas / liquid	orange	orange-red
iodine, I ₂	black solid / purple gas	brown	Purple