SH 2 Year – End Practical Examination

NATIONAL JUNIOR COLLEGE



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.

Higher 2

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.

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





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

9729/04

REGISTRATION NUMBER

SUBJECT

CHEMISTRY

Paper 4 Practical

CANDIDATE

NAME

CLASS

BLANK PAGE

1 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.

$$H^{+}(aq) + OH^{-}(aq) \rightarrow H_2O(l)$$
 ΔH_n

In this question, you will carry out a series of experiments where different volumes of **FA1** and **FA2** 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

- 1 Support the Styrofoam cup in a 250 cm³ beaker.
- 2 Using a measuring cylinder, place 20.0 cm³ of **FA 2** into the Styrofoam cup.
- 3 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.
- 5 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.
- 7 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.

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

Table 1.1

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

[1]

(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]

- (ii) 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. [1]
- (iii) From your graph, determine the maximum temperature change, ΔT_{max} , and the volume of FA 2 required to obtain this.

 $\Delta T_{max} = \dots ^{\circ}C$ V_{FA2} = cm³ [1]

Calculations

- (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 $4.18 \text{ J g}^{-1} \text{ K}^{-1}$ and that its density is 1.00 g cm^{-3} .

heat evolved =

[1]

(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).

(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 ΔH_n .

Explain whether this omission could have been the reason for the discrepancy between the value obtained by Student \bf{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.
- ² To this test-tube, add about 1 cm³ of **FA 5**. You should estimate, not measure, this volume.
- ³ 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.

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.
- 5 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.
- 6 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]

Calculations

(d) Calculate the number of moles of **FA4** in the volume of **FA4** recorded in (c), and hence deduce the concentration of Cu²⁺ ions, [Cu²⁺], in **FA6**.

[Cu²⁺] in **FA 6** =[2]

(e) Calculate the concentration of CuSO₄ in **FA 3**.

the concentration of CuSO₄ in **FA 3** =

Hence, calculate the mass of CuSO₄•5H₂O crystals required to prepare a 1 dm³ solution of FA 3.

[A_r: Cu, 63.5; H, 1.0; O, 16.0; S, 32.1]

the mass of CuSO₄•5H₂O required =

[2]

Analysis and Evaluation

(f) A teacher performs this same experiment, using the quantities described earlier, and obtains a mean titre volume of 24.85 cm³.

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]

(h) Identify two different chemical processes that use iodide ions in this experiment.

.....[1]

[Total: 15]

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.

- **FA7** 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.

	Test	Observations
(i)	Add 1 cm depth of FA7 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.	

Table 3.1

(a)

iii)	Add 1 cm depth of FA 8 to a test-tube
()	Add aqueque acdium hydroxide
	Add aqueous sodium hydroxide
	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		

(b)

Table 3.2

Test	Observations		
Test	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]

9729/04/21

	Obser	vations
	FA 8	
ubes		
cm		

14

Table 3.3

7051	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:

NJC SH2 Preliminary Examination

	- \
	<u>ר</u> ו
	61
۰.	-,

Test

(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]

	Toot	Observations	
	7651	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 .	

Table 3.4

Identity of solution P:

[1]

[Total : 14]

4 Planning

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.

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

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

17

.....

.....

(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_r*: B, 10.8; *Al*, 27.0; Ga, 69.7; In, 114.8; *Tl*, 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

	reaction with			
callon	NaOH(aq)	NH₃(aq)		
aluminium, A <i>l</i> 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), 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, CO3 ²⁻	CO ₂ liberated by dilute acids	
chloride, C <i>l</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 $NH_3(aq)$)	
iodide, I⁻(aq)	gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq))	
nitrate, NO₃⁻ (aq)	NH₃ liberated on heating with OH⁻(aq) and A <i>I</i> foil	
nitrite, NO₂⁻ (aq)	NH ₃ liberated on heating with OH [−] (aq) and A <i>l</i> foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)	
sulfate, SO₄²⁻ (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, NH₃	turns damp red litmus paper blue		
carbon dioxide, CO2	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, O2	relights a glowing splint		
sulfur dioxide, SO2	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, I2	black solid / purple gas	brown	Purple