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

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 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.

Shift
Laboratory

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.

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

Presentation [1] shows appropriate units **and** 3 or 4 sf in **all** final ans in Q1 & 2.

Any calculations not attempted loses this mark.

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

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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, H_2SO_4.

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.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l) \qquad \Delta H_n$$

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**

1 Support the Styrofoam cup in a 250 cm^3 beaker.

2 Using a measuring cylinder, place 20.0 cm^3 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 cm^3 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 cm^3.

Table 1.1

Experiment	**1**	**2**	**3**	**4**	**5**	**6**
Volume of **FA 2** / cm^3	20.0	30.0	40.0	50.0	33.0	36.0
Volume of **FA 1** / cm^3	40.0	30.0	20.0	10.0	27.0	24.0

[1] Total volume for expt 1 to 6 = 60.0 cm^3 and vol of FA 2 for expt 5 and 6 chosen between 30.0 to 40.0 cm^3

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

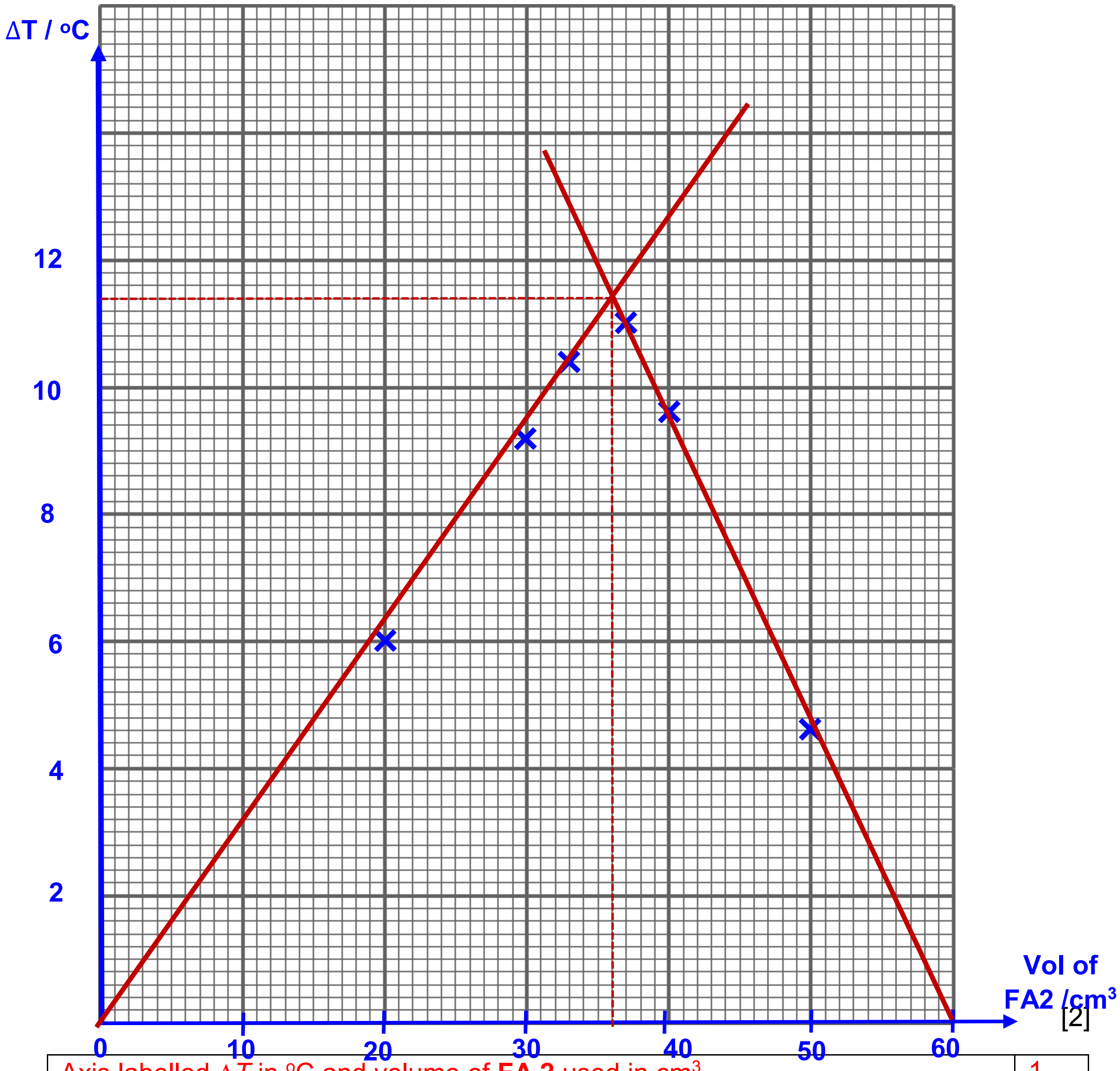
Experiment	Initial Temperature, $T_{initial}$ / °C	Maximum Temperature, T_{max} / °C	ΔT/ °C
1	28.4	34.4	+6.0
2	28.4	37.6	+9.2
3	28.4	38.0	+9.6
4	28.4	33.0	+4.6
5	28.4	38.8	+10.4
6	28.4	39.4	+11.0

[1] Clear header with units **[1]** All recorded values to 1 d.p.

[2]

(b) **(i)** On the grid given below, plot a graph of Δ***T*** (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, **Δ*T*$_{max}$.**



[2]

Axis labelled Δ*T* in °C and volume of **FA 2** used in cm^3 Linear scales chosen to use more than half of each axis	1
All points recorded (for 6 experiments). Correct plotting – each point accurately plotted (within 1⁄2 small square and in the correct square).	1

(ii) Given that **Δ*T*** = 0 at volume of **FA 2** = 0.0 and 60.0 cm^3, draw two straight lines of best fit. The first best fit line should be drawn using the plotted points where **Δ*T*** is increasing and the second best fit line should be drawn using the plotted points where **Δ*T*** is decreasing. Extrapolate these two lines until they cross. [1]

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

Δ*T*$_{max}$ = ……… +11.4 ……… °C

V$_{FA2}$ = ……… 36.0 ……… cm^3

[1]

Calculations

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

(i) calculate the concentration of NaOH in **FA 2**

$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$

Using $V_{FA2} = 36.0\ cm^3$

volume of H_2SO_4 reacted = 60.0 – 36.0 = 24.0 cm^3

Amount of H_2SO_4 reacted = 24.0/1000 x 1.00 = 0.0240 mol

Amount of NaOH reacted = 0.0240 x 2 = 0.0480 mol

Concentration of NaOH = $\frac{0.0480}{36.0}$ x 1000 = 1.333 = 1.33 mol dm^{-3}

concentration of NaOH in **FA 2** = 1.33 mol dm^{-3} ……………………………………….

[2]

(ii) calculate the heat evolved at ΔT_{max}.

You may assume that the specific heat capacity of the reaction mixture is 4.18 J g^{-1} K^{-1} and that its density is 1.00 g cm^{-3}.

heat evolved = 60.0 x 4.18 x 11.4 = 2859.12 = 2860 J

heat evolved = 2860 J ……………………………….

[1]

(d) Using your answer in **(c)(ii)**, calculate the enthalpy change of neutralisation, ΔH_n.

Amount of water produced = amount of NaOH reacted = 0.0480 mol

$\Delta H_n = -\frac{2859.12}{0.0480} = -59565\ J\ mol^{-1} = -59.6\ kJ\ mol^{-1}$

ΔH_n = – 59.6 kJ mol^{-1} ……………………………………

[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 ΔT_{max}: The magnitude of ΔT_{max} decrease **[1]** ……………………………………………………………..…………………...

explanation: Ethanedioic acid is a dibasic weak acid and does not dissociate completely in aqueous solution.

……………… Energy will be required to further ionise the unionised ethanedioic acid. **[1/2]**

……………… Hence, for the same mass of solution and same amount of H_2O formed, the amount of heat change will be smaller / lesser heat evolved. **[1/2]**

…………………………………………………………………………………………………

…………………………………………………………………………………………………

…………………………………………………………………………………………………

[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 ΔH_n.

Explain whether this omission could have been the reason for the discrepancy between the value obtained by Student **A** and the one published.

Omission of the specific heat capacity of the cup **could not [1]** have been the reason for the discrepancy because this would result in a **less exothermic ΔH_n [1/2]** since the cup would also absorb heat. **[1/2]**

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

[2]

[Total: 15]

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

FA 3 is aqueous copper(II) sulfate, $CuSO_4(aq)$, of unknown concentration.

FA 4 is 0.100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.

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 cm^3 of this diluted solution would require approximately 25 cm^3 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 plastic dropper, add 5 drops of **FA 3** to a test-tube.
2. To this test-tube, add about 1 cm^3 of **FA 5**. You should estimate, not measure, this volume.
3. Use another dropper 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 = 32 ....

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

Dilution factor = 32 ÷ 5 = 6.4 times

Vol of **FA 3** to use = 250 ÷ 6.4 = 39.06 cm^3 [1]

[1] correctly calculates volume based on previous answer

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 cm^3 and 43.00 cm^3 of **FA 3** into the 250 cm^3 graduated flask labelled **FA 6**.

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

Final burette reading / cm^3	42.75
Initial burette reading / cm^3	0.00
Volume of **FA 3** added / cm^3	42.75

[1] follows instructions of using between 42.50 cm^3 and 43.00 cm^3 of **FA 3**

[1]

Make up the contents of the flask to the 250 cm^3 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 cm^3 of **FA 6** into a conical flask.
3. Use the measuring cylinder to add 10.0 cm^3 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.

[1] Tabulates initial & final burette readings and vol added in **both** tables, with correct units (do not award mark if 50 is used as initial burette reading)

[1] All burette readings are recorded to 2 d.p and has at least 2 titres within ±0.10 cm^3

[2] Accuracy: **[2]** if student's mean is ±0.3 cm^3 from teacher's mean

[1] if student's mean is ±0.5 cm^3 from teacher's mean

Titration number	1	2
Final burette reading / cm^3	23.40	23.50
Initial burette reading / cm^3	0.00	0.00
Volume of **FA 4** added / cm^3	23.40	23.50

[4]

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

Average volume of **FA 4** used = $\frac{23.40 + 23.50}{2}$ = 23.45 cm^3

[1] correct calculation, do not award this mark if no calculations shown, if titres used are not 'ticked' in the table (tick only if there are 3 titres), if there are arithmetic errors in the table

[1]

Calculations

(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**.

[1] calculating moles of **FA 4** used

[1] final answer (allow ecf)

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

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

No of moles of **FA 4** used = $\frac{23.45}{1000}$ x 0.100 = 2.345 x 10^{-3} = No of moles of Cu^{2+} in 25.0 cm^3

No of moles of Cu^{2+} in 250 cm^3 = 2.345 x 10^{-2}

$[Cu^{2+}]$ in **FA 6** = $\frac{2.345 \times 10^{-2}}{250}$ x 1000 = 0.0938 mol dm^{-3}

$[Cu^{2+}]$ in **FA 6** = ……………………………….

[2]

(e) Calculate the concentration of $CuSO_4$ in **FA 3**.

No of moles of Cu^{2+} in 42.75 cm^3 = 2.345 x 10^{-2}

$[Cu^{2+}]$ in **FA 3** = $\frac{2.345 \times 10^{-2}}{42.75}$ x 1000 = 0.5485

= 0.549 mol dm^{-3}

[1] final answer (allow ecf)

the concentration of $CuSO_4$ in **FA 3** = ……………………………….

Hence, calculate the mass of $CuSO_4.5H_2O$ 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]

M_r of $CuSO_4.5H_2O$ = 249.6

Mass of $CuSO_4.5H_2O$ = 0.5485 x 249.6 = 136.91 = 137 g

[1] final answer (allow ecf)

the mass of $CuSO_4.5H_2O$ 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 cm^3.

The errors (uncertainties) associated with each reading using a graduated flask, pipette and burette are, ±0.15 cm^3, ±0.1 cm^3 and ±0.05 cm^3 respectively.

Calculate the maximum total percentage error (uncertainty) of this mean titre volume.

Total % error = $(\frac{\pm 0.15}{250} \times 100) + (\frac{\pm 0.1}{25.0} \times 100) + (\frac{\pm 0.05 \times 2}{24.85} \times 100)$

= ±0.862%

[1] accept if % error in measuring 42.50 – 43.00 cm^3 of **FA 3** with a burette is included in the total error calculation

Do not award mark if ± is missing.

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]

M_r of KI = 166.0

No of moles of I^- used = $\frac{90}{166.0} \times \frac{10}{1000}$ = 5.42×10^{-3} **[1/2]**

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

No of moles of I^- is still in excess (since > 2 times the amt of Cu^{2+}; 2.345×10^{-3}) **[1/2]**

hence accuracy of her titre volumes is not affected. **[1] ecf**

………

Do not award any mark if student made no attempt to calculate amt of I^- for comparison purpose. [2]

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

Reduction of Cu^{2+} (to Cu^+). …. [½] for each chemical process ………………..

Precipitation of CuI (from Cu^+ and I^-). …. ……………[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.

- **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.	No observable change OR colourless solution OR no precipitate formed. **[½]**
	Transfer the mixture into a **boiling tube** and **carefully** warm the boiling tube and its contents. Do **not** heat until the mixture boils.	On warming, colourless, pungent gas evolved turned moist red litmus paper blue. **[½]**
(ii)	Add 1 cm depth of **FA 8** to a test-tube. Add aqueous ammonia slowly, with shaking, until no further change is seen.	White precipitate formed which is soluble in excess NH_3(aq) to give a colourless solution. **[½]**

(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.	White precipitate formed which is soluble in excess NaOH(aq) to give a colourless solution. **[½]**

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	NH_4^+ **[1]**	Zn^{2+} **[1]**

[4]

(b) **Table 3.2**

Test	*Observations*	
	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).	Purple $KMnO_4$ remains **[½]**	Purple $KMnO_4$ remains **[½]**

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**: SO_4^{2-} **[½]** NO_3^- **[½]**……………………

Possible anions present in **FA 9**: SO_4^{2-} **[½]** NO_3^- **[½]**…………………..… [3]

(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.	White precipitate formed which is soluble in excess NaOH(aq).	No observable change.
warm the mixture gently and carefully then add a piece of aluminium foil to the mixture.	No observable change OR no gas evolved. **[½]**	Colourless, pungent gas evolved turned moist red litmus paper blue. **[½]**

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	SO_4^{2-} **[1]**	NO_3^- **[1]**

[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: Add aqueous silver nitrate, followed by aqueous ammonia in excess…………..

Table 3.4

Test	*Observations*
Add 1 cm depth of **FA 7** to a test-tube. Add aqueous silver nitrate, with shaking. **[½]** Add aqueous ammonia, with shaking, until no further change is seen. **[½]**	White precipitate observed. **[½]** White precipitate soluble in excess aqueous NH_3 to give a colourless solution.**[½]**

Halide ion present in **FA 7**: chloride…………………..**[1]**

[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*
		solution P
(i)	Add aqueous ammonia to 1 cm depth of **solution P**, until in excess.	No observable change. **(Ca^{2+}/Ba^{2+}/NH_4^+)**
(ii)	Add aqueous sodium hydroxide to 1 cm depth of **solution P**, until in excess. 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 observable change. **(Ca^{2+}/Ba^{2+}/NH_4^+)** No gas evolved. **(Not NH_4^+)**
(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**. **(CO_2 forms white ppt with limewater $Ca(OH)_2$)** **$Ca(OH)_2 (aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)$**

Identity of **solution P**: $Ca(OH)_2$ / $Ba(OH)_2$ **[1]**………………... [1]

[Total : 14]

Do not award this mark if student only identified the cation Ca^{2+} / Ba^{2+} / limewater

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•16H_2O by heating the salt until it becomes anhydrous.

The equation for the dehydration of the hydrated Group 13 sulfate salt, X•16H_2O, is as shown.

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

FB 1 is the hydrated salt, X•16H_2O.

(a) Plan an investigation to identify the metal in a hydrated Group 13 sulfate salt, X•16H_2O 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
- how you would ensure that the salt becomes anhydrous

[5]

Procedure

1. Weigh a crucible with its lid and record the mass.
2. Add 2.0 g of **FB 1** to the crucible and weigh the crucible and lid with **FB 1** and record the mass.
3. (Place the crucible on the pipe-clay triangle.) Heat the crucible and contents gently for approximately two minutes with the lid on.
4. Remove the lid. Then heat the crucible and contents strongly for approximately four minutes.
5. Replace the lid and leave the crucible and residue to cool for at least five minutes.
6. Reweigh the crucible and contents with the lid on. Record the mass.
7. Remove the lid. Heat the crucible and contents strongly for a further two minutes.
8. Replace the lid and leave the crucible and residue to cool for at least five minutes. Then reweigh the crucible and contents with the lid on. Record the mass.
9. Repeat the heat-cool-weigh process until mass of residue is constant (± 0.01 g). Record all the masses.

Sample Table:

Mass of empty crucible and lid	/ g	**A**
Mass of crucible and lid and **FB 1** used	/ g	**B**
Mass of crucible and lid and residue after first heating	/ g	**C**
Mass of crucible and lid and residue after second heating	/ g	**D**
Mass of crucible and lid and residue after third heating	/ g	**D**
Mass of **FB 1** used	/g	**B – A**
Maximum mass loss	/g	**B – D**

*** Mass of FB 1 used & Maximum mass loss are optional in Sample Table**

[1] logical sequence of steps for experimental procedure

(weigh → heat → cool → reweigh)

[1/2] gentle heating at first (to prevent sudden expansion of air trapped in solid)

[1/2] heat with lid on at first, subsequent heating without lid (to allow water vapour to escape)

[1/2] allow crucible and contents to cool with lid on (to avoid contact with moisture in air), before reweighing

[1] repeat heat-cool-weigh process until consistent mass of ±0.01 g obtained

[1] sample table with alphabets representing masses

(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.

$X\cdot16H_2O(s) \rightarrow X(s) + 16H_2O(g)$

Calculate the no of mole of water = mass lost (**B – D**) / 18 = **E**

Calculate the no of mole of anhydrous residue = **E** / 16 = **F** **[1]**

Calculate relative formula mass of X = mass of residue (**D– A**) / **F** **[1]**

[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; O: 16.0; A*l*, 27.0; S, 32.1; Ga, 69.7; In, 114.8; T*l*, 204.4]

Given that X is a Group 13 sulfate salt, the formula of X must be $M_2(SO_4)_3$.

A_r of Metal = [352.5 – 3(32.1 + 4x16.0)] / 2
= 32.1 (1 d.p) **[1]**

Metal in X is Aluminium. **[1]**

[2]

(d) Suggest why using a crucible is better than a boiling tube in this experiment.

Using a crucible allows water of crystallization to escape more easily / greater surface area for the water of crystallization to escape.

Water of crystallization may condense on the upper cooler part of the boiling tube and flow back to the hot part of residue, causing the boiling tube to crack.

....

....

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[Total: 10]

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