



Catholic Junior College

JC2 Preliminary Examinations

Higher 2

CANDIDATE
NAME

CLASS

2T

CHEMISTRY

9729/04

Paper 4 Practical

26 August 2025
2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name and class in the boxes above.

Give details of the practical shift and laboratory, where appropriate, in the boxes provided.

Write in dark 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 17 and 18.

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	/ 13
2	/ 11
3	/ 19
4	/ 12
Total	/ 55

This document consists of **18** printed pages.

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

1 Investigation of an organic compound and an inorganic compound

FA 1 is an aqueous solution that contains a mixture of salts with two cations and one anion listed in the Qualitative Analysis Notes.

FA 2 is an aqueous solution of an organic compound, **X**, with only one functional group present.

You will perform tests to identify:

- the ions present in **FA 1**.
- the functional group of **X** in **FA 2**.

(a) (i) Carry out the following tests. Carefully record your observations in Tables 1.1 and 1.2.

The volumes given below are approximate and should be estimated rather than measured.

Test and identify any gases evolved.

If there is no observable change, write **no observable change**.

Table 1.1

tests		observations
1	To 1 cm depth of FA 1 in a boiling tube, add aqueous sodium hydroxide until there is no further change. Heat the boiling tube gently.	<u>Green ppt</u> formed, turned <u>brown on contact with air</u> , <u>insoluble</u> in excess NaOH (aq). <u>Gas evolved on heating turned moist red litmus paper blue. NH₃ (g) evolved.</u>
2	To 1 cm depth of FA 1 in a test-tube, add a few drops of Ba(NO ₃) ₂ (aq). Then, add excess nitric acid.	<u>White ppt</u> formed, <u>insoluble</u> in excess HNO ₃ .
3	To 1 cm depth of FA 1 in a test-tube, add excess aqueous hydrogen peroxide until no further change.	Pale green solution turned <u>brown/ orange-yellow</u> . <u>Effervescence observed. O₂ gas relighted a glowing splint.</u>

From your observations, identify the ions present in **FA 1**.

The cations are **Fe²⁺** and **NH₄⁺**

The anion is **SO₄²⁻**

[7]

- (ii) Explain the observation for the reaction between **FA 1** and aqueous hydrogen peroxide in Test 3.

In Test 3, H_2O_2 oxidises Fe^{2+} to Fe^{3+} which explains for the change in colour of the solution from pale green to brown/ orange-yellow.

[1]

Table 1.2

tests		observations
1	To 1 cm depth of FA 2 in a test tube, add 2 cm of depth of water. Test this solution with Universal Indicator paper. Hence conclude the pH of the resultant solution of FA 2 . Keep this solution for test 2 below.	No observable change <u>UI paper turns red.</u> <u>pH = 1 or 2</u>
2	To the resultant solution in test 1, add 1 full spatula of solid Na_2CO_3 .	<u>Effervescence observed</u> <u>Gas evolved forms white ppt with limewater.</u> <u>$\text{CO}_2(\text{g})$ evolved.</u>
3	To a 1 cm depth of FA 2 in a test-tube, add 1 cm depth of aqueous silver nitrate.	An immediate <u>white precipitate of AgCl</u> is obtained.

[3]

- (b) (i) Use your results of Test 3 in Table 1.2 to identify the anion present in **FA 2**.

Anion present: Cl^-

- (ii) Suggest a possible functional group that could be present in **X** in **FA 2** and state the evidence by completing Table 1.3.

Table 1.3

Functional group	evidence
Acyl chloride.	FA 2 reacts with water to form an <u>acidic solution</u> and it also reacts with <u>aqueous AgNO_3 to form white ppt of $\text{AgCl}(\text{s})$.</u>

[2]

[Total: 13]

[Turn over

2 Determination of the amount of water of crystallisation in $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$.

In this experiment, you are to determine the amount of water of crystallisation (value of n) in a sample of hydrated copper(II) sulfate, $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$ by titration.

When excess aqueous potassium iodide is added to the aqueous Cu^{2+} ions, iodine and a white precipitate of copper(I) iodide are produced.



The amount of iodine produced is found by titration with aqueous thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$.



You are provided with

FA 3 is a solution of aqueous hydrated copper(II) sulfate of concentration 20.0 g dm^{-3} .

FA 4 is a solution of aqueous potassium iodide, KI.

FA 5 is $0.100 \text{ mol dm}^{-3}$ aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

Starch indicator

(a)(i) Titration of **FA 3** against **FA 5**

1. Fill a burette with **FA 5**.
2. Use a pipette to transfer 25.0 cm^3 of **FA 3** into a 250 cm^3 conical flask.
3. Use a measuring cylinder to add about 15 cm^3 of **FA 4** to this flask. A white precipitate forms in a brown solution.
4. Run **FA 5** from the burette into this flask. Near the end-point, when the brown solution becomes pale, add about 1 cm^3 of starch indicator.
5. Continue adding **FA 5** slowly. The end-point is reached when the **solution** first becomes colourless. The white precipitate remains.
6. Record your titration results, to an appropriate level of precision, in the space provided below.
7. Repeat points 2 to 6 until consistent results are obtained.

Titration Results

	1	2
Final burette reading / cm^3	20.00	40.00
Initial burette reading / cm^3	0.00	0.00
Volume of FA 5 used / cm^3	20.00	20.00

✓

✓

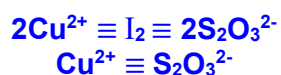
[3]

- (ii) From your titrations, obtain a suitable volume of **FA 5**, V_{FA5} , to be used in your calculations. Show clearly how you obtained this volume and place a tick (✓) under the readings used.

$$\text{average titre} = \frac{20.00 + 20.00}{2} = 20.00 \text{ cm}^3$$

$$V_{\text{FA5}} = \frac{20.00 \text{ cm}^3}{[3]}$$

- (b) (i) Calculate the concentration of $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$ in **FA 3**.



25.0 cm³ of FA 3 (Cu^{2+}) reacts with 20.00 cm³ of FA 5 ($\text{S}_2\text{O}_3^{2-}$)
amt of Cu^{2+} in 25.0 cm³ of FA 3 = amt of $\text{S}_2\text{O}_3^{2-}$ in 20.00 cm³ of FA 5

$$\begin{aligned} &= 0.100 \times \frac{20.00}{1000} \\ &= 2.00 \times 10^{-3} \text{ mol} \end{aligned}$$

$$\begin{aligned} \text{conc of CuSO}_4 \text{ in FA 3} &= \frac{1000}{25.00} \times 2.00 \times 10^{-3} \\ &= 0.0800 \text{ mol dm}^{-3} \end{aligned}$$

$$\text{concentration of CuSO}_4 \cdot n\text{H}_2\text{O} = \frac{0.0800 \text{ mol dm}^{-3}}{[1]}$$

[Turn over

- (ii) Determine the relative molecular mass of $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$ in **FA 3** and hence calculate the value of n . You **must** show your working. [Ar: H, 1.0; O, 16.0; S, 32.1; Cu, 63.5]

Given that FA 3 contains 20.0 g dm^{-3} of $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$.

mass of 0.0800 mol of $\text{CuSO}_4 \cdot n\text{H}_2\text{O} = 20.0 \text{ g}$

mass of 1 mol of $\text{CuSO}_4 \cdot n\text{H}_2\text{O} = \frac{20.0}{0.0800} = 250.0 \text{ g}$

M_r of $\text{CuSO}_4 \cdot n\text{H}_2\text{O} = 250.0$

relative molecular mass, M_r , of $\text{CuSO}_4 \cdot n\text{H}_2\text{O} = \text{250.0}$

M_r of $\text{CuSO}_4 \cdot n\text{H}_2\text{O} = 250.0$

$63.5 + 32.1 + 4(16.0) + n(2(1.0) + 16.0) = 250.0$

$159.6 + 18n = 250.0$

$$n = \frac{90.4}{18}$$

$$= 5.02$$

$$= 5 \text{ (whole number)}$$

value of $n = \text{5}$
[2]

- (c) Calculate the maximum total percentage uncertainty for your titration in **2(a)(i)** if the uncertainty associated with each reading using a 25.0 cm^3 pipette and a burette are $\pm 0.06 \text{ cm}^3$ and $\pm 0.05 \text{ cm}^3$ respectively.

$$\% \text{ uncertainty due to pipette} = \pm \frac{0.06}{25.0} \times 100\% = \pm 0.240\%$$

$$\% \text{ uncertainty due to burette used in titration} = \pm \frac{2 \times 0.05}{20.00} \times 100\% = \pm 0.500\%$$

$$\text{Maximum total percentage uncertainty} = 0.240 + 0.500 = \pm 0.740\% \text{ (3 s.f.)}$$

Percentage uncertainty = $\pm 0.740\%$
[2]

[Total: 11]

3 Determination of an enthalpy change of neutralisation by thermometric titration

FA 6 is 1.00 mol dm^{-3} aqueous sodium hydroxide, NaOH.

FA 7 is aqueous sulfuric acid, H_2SO_4 .

You are to carry out a thermometric titration to determine the enthalpy change of neutralisation per mole of water formed when these two solutions, **FA 6** and **FA 7** react. This involves adding volumes of aqueous sulfuric acid to a fixed volume of aqueous sodium hydroxide and measuring the temperature of the resulting solution.

(a) **Before starting any practical work, read through the instructions carefully and complete the table in the space provided on page 8.** Record, to the appropriate level of precision:

- all volumes of acid, **FA 7** added, V
- the maximum temperature, T , reached after each addition of **FA 7**

It is important that the volume of **FA 7** recorded is the total volume you have added up to the point when the temperature reading was made.

Note: If you overshoot on an addition, record the actual volume of **FA 7** added up to that point.

Procedure

1. Place a polystyrene cup inside a second polystyrene cup and place both cups in a 250 cm^3 beaker. The retort clamp provided may be used to clamp the beaker to prevent it from tipping.
2. Use the pipette labelled **FA 6** to transfer 25.0 cm^3 of **FA 6** into the first polystyrene cup.
3. Fill the burette labelled **FA 7** with **FA 7**.
4. Stir the **FA 6** in the cup gently with the thermometer. Read and record its temperature.
5. Use the burette to add 2.00 cm^3 of **FA 7** to the cup and stir the mixture gently with the thermometer. Read and record both the maximum temperature and the actual total volume of **FA 7** added.
6. Repeat step 5 until a total of 24.00 cm^3 of **FA 7** has been added. For each addition of **FA 7**, read and record both the maximum temperature and the actual total volume of **FA 7** added up to that point.

[Turn over

Results

Total volume of FA 7 added, V /cm³	Max Temp after addition of FA 7, T /°C
0.00	29.2
2.00	31.2
4.00	33.0
6.00	34.4
8.00	35.6
10.00	36.8
12.00	37.8
14.00	37.0
16.00	36.2
18.00	35.6
20.00	35.0
22.00	34.6
24.00	34.2

[3]

- (b) Plot a graph of temperature, T , on the y -axis, against total volume of **FA 7**, V , on the x -axis on the grid in Fig 3.1. Your scale on the y -axis should allow for extrapolation above the highest temperature recorded.

Draw two lines of best-fit, taking into account the points when the temperature of the mixture was rising and the points when the temperature was falling. Each line should have a shape best suited to its plotted points.

Extrapolate (extend) both lines until they intersect.

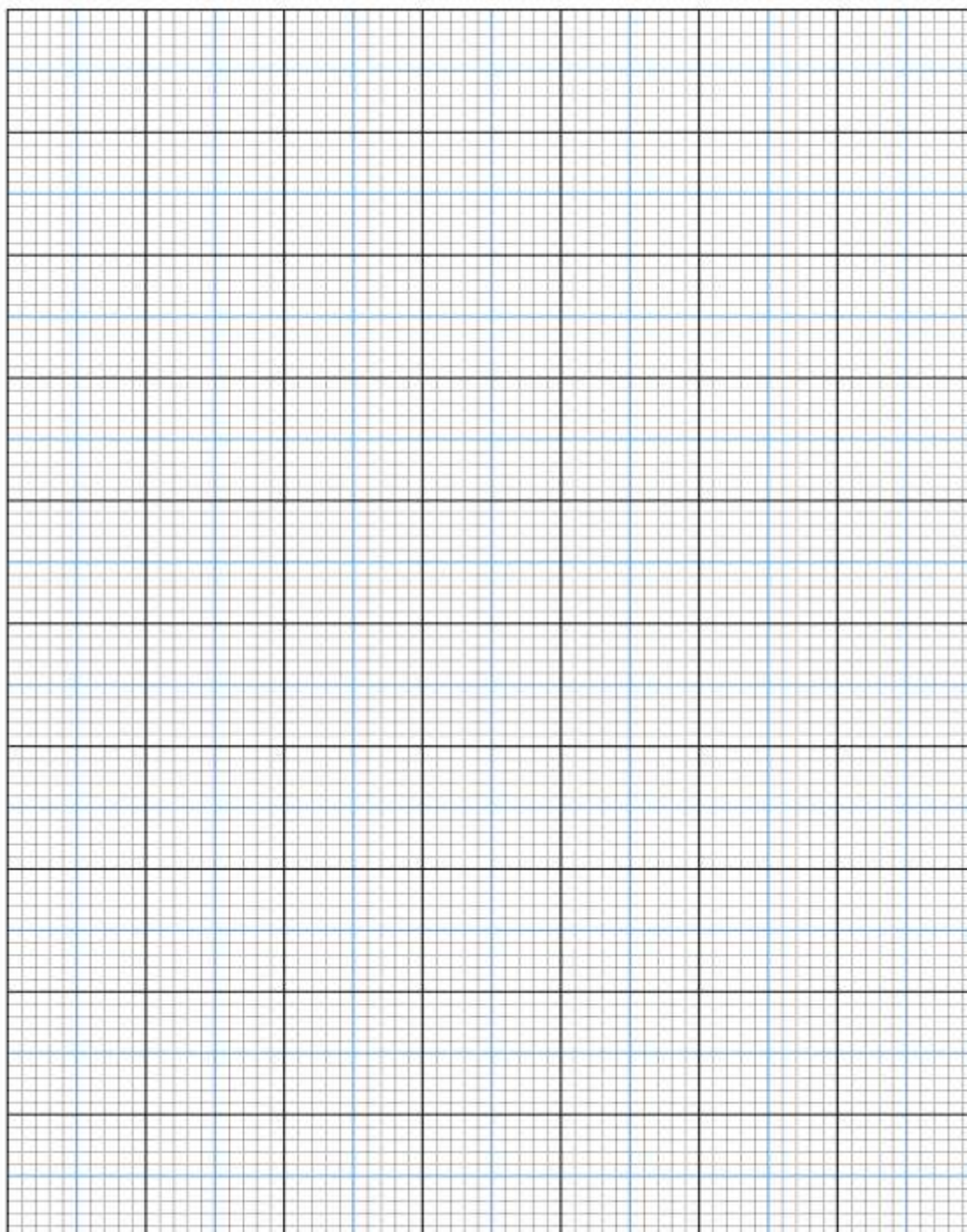
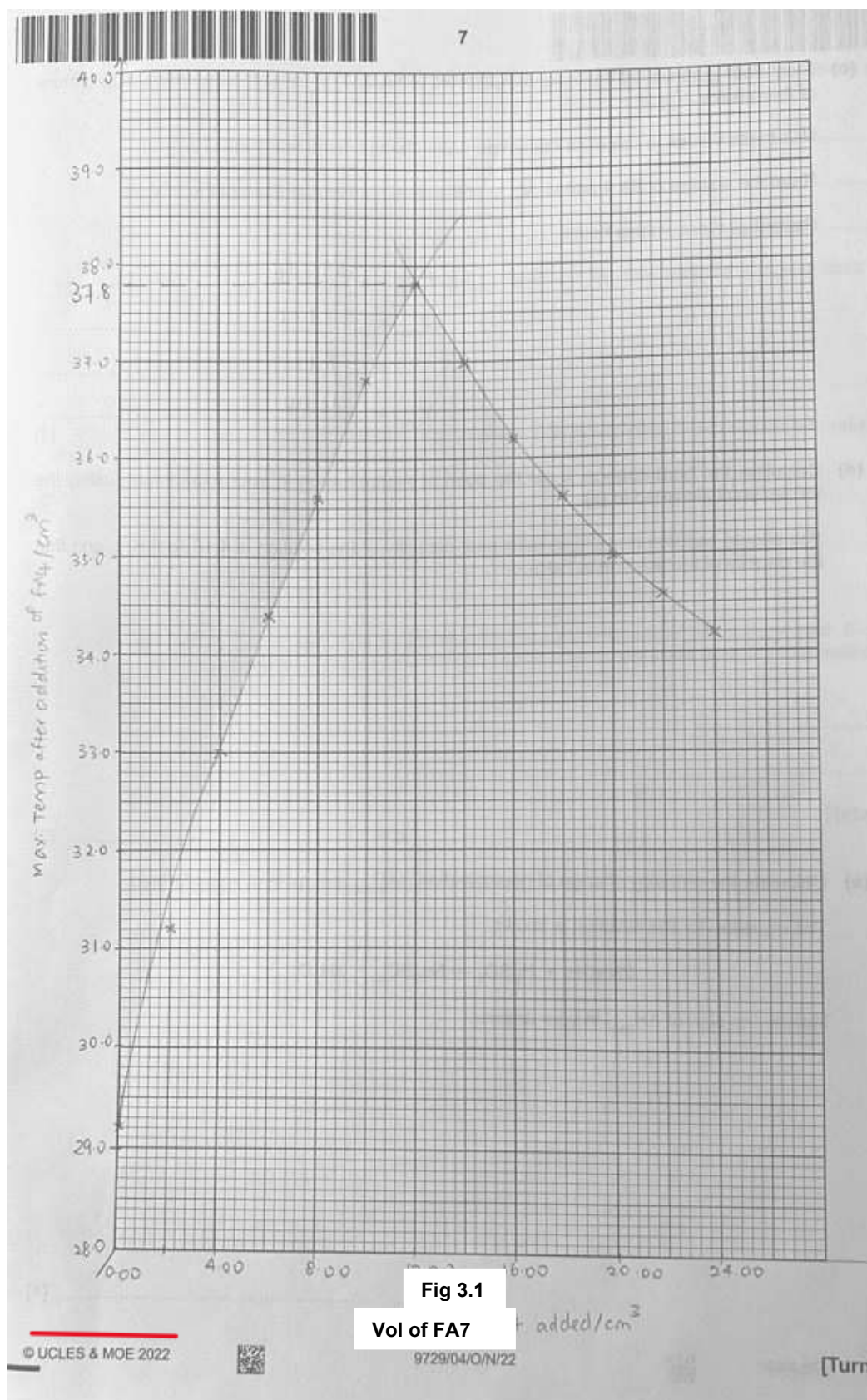


Fig. 3.1

[3]

[Turn over



- (c) From your graph, read the initial temperature of **FA 6**, T_{initial} , and the maximum temperature of the mixture, T_{max} .

Use these values to calculate the temperature change in the reaction, ΔT .

Read the volume of **FA 7** added, V_{neut} , at the maximum temperature of the mixture.

Record all these values below.

$$\begin{aligned} T_{\text{initial}} &= \underline{T_{\text{initial}} = 29.2\text{ }^{\circ}\text{C}} \\ T_{\text{max}} &= \underline{T_{\text{max}} = 37.8\text{ }^{\circ}\text{C}} \\ \Delta T &= \underline{\Delta T = 8.6\text{ }^{\circ}\text{C}} \\ V_{\text{neut}} &= \underline{V_{\text{neut}} = 12.00\text{ cm}^3} \end{aligned}$$

[4]

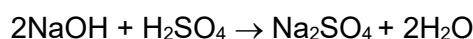
- (d) Calculate the heat change, q , at the point of neutralisation in your experiment, using the values you calculated in (c).

You should assume that the specific heat capacity of the solution is $4.18\text{ J g}^{-1}\text{ K}^{-1}$, and that the density of the solution is 1.0 g cm^{-3} .

$$\begin{aligned} \text{Heat change, } q &= mc\Delta T = (25+12)(4.18)(8.6) \\ &= \underline{1330\text{ J}} \end{aligned}$$

$$q = \underline{1330\text{ J}} \quad [2]$$

- (e) Calculate the enthalpy change of neutralisation, ΔH_{neut} , per mole of water formed. The equation for the reaction is shown.



Include the sign of ΔH_{neut} in your answer.

$$\text{Amount of NaOH} = (1)\left(\frac{25}{1000}\right) = 0.0250\text{ mol}$$

Since $\text{H}_2\text{O} \equiv \text{NaOH}$,

$$\text{amount of H}_2\text{O} = \underline{0.0250\text{ mol}}$$

$$\begin{aligned} \text{Enthalpy change of neutralisation, } \Delta H_{\text{neut}} &= -\frac{q}{\text{moles of water}} \\ &= -\frac{1330}{0.0250} \\ &= \underline{-53200\text{ J mol}^{-1}} \\ &= \underline{-53.2\text{ kJ mol}^{-1}} \end{aligned}$$

$$\Delta H_{\text{neut}} = \underline{-53.2\text{ kJ mol}^{-1}} \quad [4]$$

[Turn over]

- (f) (i) Suggest why the magnitude of ΔH_{neut} obtained by this method is lower than the magnitude of the true ΔH_{neut} .

This method of obtaining the ΔH_{neut} would have heat loss to the surroundings and/or the polystyrene cup.

.....[1]

- (ii) Suggest **two** improvements to the method that would give a more accurate value for ΔH_{neut} .

1. Do this experiment in a draught-free environment.

2. Cover the plastic cup with a lid to reduce heat loss.

.....[1]

- (g) Your value of V_{neut} could be used to calculate the concentration of the sulfuric acid. Explain why a conventional titration using an indicator gives a more accurate volume of neutralisation for this calculation.

A conventional titration will give a more accurate volume of neutralisation because this volume can be obtained directly from the burette readings at the end-point and the burette readings can be read precisely to $\pm 0.05 \text{ cm}^3$.

.....[1]

[Total: 19]

4 Planning – Preparation of paracetamol

Paracetamol, $\text{HOC}_6\text{H}_4\text{NHCOCH}_3$, is a white solid at room temperature. It is made by the reaction of 4-aminophenol, $\text{HOC}_6\text{H}_4\text{NH}_2$, with ethanoic anhydride, $(\text{CH}_3\text{CO})_2\text{O}$. The equation for this reaction is shown.



1.1 g of 4-aminophenol was added into 3 cm³ of water. 1.5 cm³ (excess) of ethanoic anhydride was added and the mixture was swirled thoroughly. The mixture was then warmed under reflux for 10 minutes, with the temperature not being allowed to raise above 70 °C.

The mixture was allowed to cool for a few minutes and then poured into 25 cm³ of cold water, stirring well to precipitate the impure solid paracetamol. The impure paracetamol was separated by filtration and then purified.

Table 4.1 gives information about some of the chemicals involved.

Table 4.1

chemical	M_r	melting point/ °C
4-aminophenol	109.0	185
ethanoic anhydride	102.0	–73
paracetamol	151.0	169

You are required to plan a procedure to prepare a sample of **10.0 g** of paracetamol using the above method.

- (a) Calculate the mass of 4-aminophenol needed to give an expected yield of 10.0 g of paracetamol, assuming the percentage yield in the whole process is 75 %.

Since 75% yield is equivalent to 10.0 g paracetamol.

for 100% yield, mass of minimum paracetamol expected = $\frac{100}{75} \times 10.0 = \underline{13.33 \text{ g}}$

amount of paracetamol = $\frac{13.33}{151.0} = \underline{0.0883 \text{ mol}}$

Since paracetamol \equiv 4-aminophenol

\therefore minimum mass of 4-aminophenol needed = 0.0883×109.0
 = 9.62 g

Mass of 4-aminophenol = 9.62 g g
 [2]

[Turn over

- (b) Plan a procedure for the preparation of 10.0 g of pure and dry paracetamol using 4-aminophenol and ethanoic anhydride.

In your plan, using 30 cm³ of water, you should include details of:

- a calculation to obtain an approximate volume of ethanoic anhydride to be used
- the apparatus you would use
- a diagram of the apparatus you would use to warm the mixture under reflux

You may assume that you have access to the following apparatus:

- reflux condenser
- water bath
- 250 cm³ round-bottomed flask
- electronic mass balance reading to 2 decimal places
- Buchner funnel with a vacuum filtration set-up
- Infra-red lamp,
- any other apparatus commonly found in a school laboratory

Procedure:

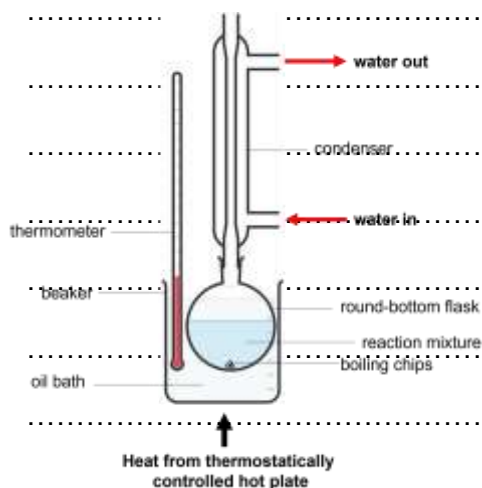
Estimated volume of ethanoic anhydride required = $\frac{9.62}{1.1} \times 1.5$

$$= 13.1 \text{ cm}^3 \approx 14 \text{ cm}^3$$

1. Weigh out accurately about 9.62 g of 4-aminophenol using an electronic mass balance on dry weighing paper. Carefully transfer to a 250 cm³ round-bottomed flask.

2. Using a 50 cm³ measuring cylinder, measure 30 cm³ of water and add to the flask. Swirl the flask.

3. Using a 25 cm³ measuring cylinder, measure 14 cm³ of ethanoic anhydride and add to the flask. Swirl the flask thoroughly.



[1; appropriate reactants and solvent used]

4. Assemble the apparatus as shown above for heating under reflux. Gently heat the flask in a thermostatically controlled boiling water/oil bath to 70 °C for about 10 minutes.
5. Using a 25 cm³ measuring cylinder, measure 25 cm³ of cold water and add into a 250 cm³ beaker.
6. Remove the contents of the flask from the hot water/oil bath and pour the contents of the flask into the 25 cm³ of cold water prepared in step 5. Stir the contents in the beaker well until crystals of impure solid paracetamol no longer form.
7. Set up a vacuum filtration apparatus. Wet the filter paper in the Buchner funnel with 1-2 cm³ of distilled water. Turn on the water aspirator. Decant the reaction mixture onto the filter paper, minimising any transfer of the solid paracetamol.
8. Add 15 cm³ of cold water to the flask. Pour the solution mixture and the crystals of paracetamol onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete. Wash the paracetamol crystals on the filter paper with 10 cm³ of ice water. Maintain the vacuum to dry the crystals as best as possible.

[7]

[Turn over

- (c) Describe the process how you would carry out recrystallisation of paracetamol in (b) to ensure you obtain a pure and dry sample of paracetamol.

Recrystallisation of Paracetamol:

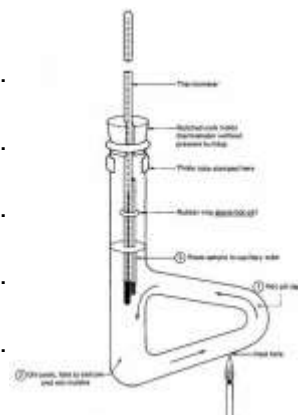
[Note: The process of recrystallisation has to be mentioned clearly with essential details.]

1. Place the paracetamol crystals in a 100 cm³ beaker. Add 50 cm³ of water.
2. Warm the mixture in a 60 °C water bath, until all the paracetamol crystals dissolves.
3. Remove the beaker from the heat, and set it aside to cool slowly in an ice/water bath. Crystals of paracetamol will form.
4. Collect the paracetamol crystals by vacuum filtration. Wash the crystals with two 10 cm³ portions of ice water. Maintain the vacuum to air dry the paracetamol crystals.
5. Transfer the dry paracetamol crystals to a pre-weighed sample container or vial.
6. Dry the crystals under an Infra-red (IR) lamp / dessicator.

[2]

- (d) Describe briefly how you would show that your sample of paracetamol is pure.

1. Fill a capillary melting point tube to a depth of 0.2 cm with the recrystallised paracetamol.
2. Place the capillary tube in the melting point apparatus (as shown in diagram). If the crystals melt sharply at 169 °C, the paracetamol prepared is pure. Otherwise, the melting point will be observed to be lower, which suggests that impurities are present.



[1]

[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

[Turn over

(b) Reactions of aqueous anions

ion	reaction
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (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 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