Surname	StemUp Model Solution	Centre Number	Candidate Number
First name(s)			2

GCE A LEVEL

A410U30-1

S24-A410U30-1

FRIDAY, 21 JUNE 2024 – MORNING

CHEMISTRY – A level component 3

Chemistry in Practice

1 hour 15 minutes

For Examiner's use only		
Question	**Maximum Mark**	**Mark Awarded**
1.	**8**	
2.	**13**	
3.	**8**	
4.	**19**	
5.	**12**	
Total	**60**	

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ADDITIONAL MATERIALS

- A calculator, pencil and ruler
- **Data Booklet** supplied by WJEC

INSTRUCTIONS TO CANDIDATES

Use black ink or black ball-point pen. Do not use gel pen or correction fluid.

You may use a pencil for graphs and diagrams only.

Write your name, centre number and candidate number in the spaces at the top of this page.

Answer **all** questions.

Write your answers in the spaces provided in this booklet. If you run out of space, use the additional page(s) at the back of the booklet, taking care to number the question(s) correctly.

INFORMATION FOR CANDIDATES

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

The maximum mark for this paper is 60.

Your answers must be relevant and must make full use of the information given to be awarded full marks for a question.

The assessment of the quality of extended response (QER) will take place in **Q2**(a)(i).

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Answer **all** questions.

1. Calcium iodate(V) is a sparingly soluble salt and can be prepared by the reaction between solutions of potassium iodate(V) and a suitable soluble calcium salt, such as calcium chloride.

$$Ca^{2+}(aq) + 2IO_3^-(aq) \longrightarrow Ca(IO_3)_2(s)$$

It is usually obtained as a hydrate, $Ca(IO_3)_2.xH_2O$, when precipitated from aqueous solution.

In acid solution, iodate(V) ions react with excess iodide ions to liberate iodine.

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \longrightarrow 3I_2(aq) + 3H_2O(l)$$

The iodine formed can be determined by titration against standard sodium thiosulfate solution.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \longrightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

Using this technique, the relative formula mass of the salt, $Ca(IO_3)_2.xH_2O$, and the value of x were determined as follows.

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Step	Method
1	An accurately weighed sample of solid $Ca(IO_3)_2.xH_2O$ was dissolved in hot water in a conical flask.
2	After cooling, excess acidified potassium iodide solution was added to the solution in the flask.
3	The solution containing the iodine (I_2) formed was titrated against 0.540 mol dm^{-3} sodium thiosulfate solution until the dark brown colour changed to pale yellow. Starch indicator was added and the titration continued until the end-point. The volume of sodium thiosulfate solution used was recorded.
4	The method was repeated with a further two samples of the solid $Ca(IO_3)_2.xH_2O$.

Results

Titration number	1	2	3
Mass of $Ca(IO_3)_2.xH_2O$ / g	0.481	0.493	0.474
Volume of $S_2O_3^{2-}(aq)$ / cm^3	21.45	22.00	21.15

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(a) For step **3**, give the colour change of the starch indicator at the end-point. [1]

blue-black to colourless

(b) Identify the titration that has the smallest percentage error in the **mass** of $Ca(IO_3)_2.xH_2O$ used. Give your reasoning. [1]

Titration number 2

0.493 g is the largest mass, so the (fixed) uncertainty of the balance is the smallest percentage of the mass.

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(c) The five steps of the calculation are shown below.

(i) Number these in the correct order. The first and last steps have been done for you. [1]

	Correct order
Convert moles of IO_3^- to moles of $Ca(IO_3)_2.xH_2O$	3
Use the balanced equation(s) to calculate the number of moles of IO_3^- ions present in the solid sample	2
Calculate the value of x in $Ca(IO_3)_2.xH_2O$	5
Convert moles of $Ca(IO_3)_2.xH_2O$ to M_r of $Ca(IO_3)_2.xH_2O$	4
Calculate the number of moles of sodium thiosulfate used in the titration	1

(ii) Carry out the calculation to determine the relative formula mass of the salt, $Ca(IO_3)_2.xH_2O$, and hence the value of x. Use the results from **titration 2** only.

You **must** show your working. [5]

$n(S_2O_3^{2-}) = 0.540 \times 22.00 \div 1000 = 0.01188$ mol

$n(I_2) = 0.01188 \div 2 = 0.00594$ mol

$n(IO_3^-) = 0.00594 \div 3 = 0.01188 \div 6 = 0.00198$ mol

$n(Ca(IO_3)_2.xH_2O) = 0.00198 \div 2 = 0.00099$ mol

$M_r(Ca(IO_3)_2.xH_2O) = 0.493 \div 0.00099 = 497.97$

$M_r(Ca(IO_3)_2) = 40.1 + 2(127 + 48.0) = 390.1$

mass of $xH_2O = 497.97 - 390.1 = 107.9$

$x = 107.9 \div 18.02 = 5.99 = 6$

$x =$ 6

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2. (a) Peroxydisulfate ions react with iodide ions to form iodine.

$$S_2O_8^{2-}(aq) + 2I^-(aq) \longrightarrow 2SO_4^{2-}(aq) + I_2(aq)$$

The rate equation for the reaction is as follows.

$$\text{rate} = k\,[S_2O_8^{2-}]\,[I^-]$$

One way of measuring the rate is to time how long it takes to produce a certain amount of iodine using the iodine clock reaction.

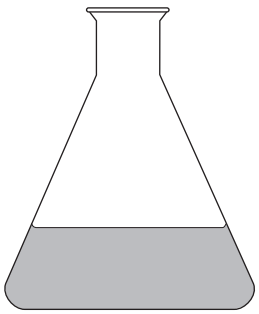
A student was asked to carry out an experiment to confirm that the reaction is first order with respect to peroxydisulfate ions.

The student was provided with the following aqueous solutions and deionised water.

0.1 mol dm^{-3} potassium peroxydisulfate
0.3 mol dm^{-3} potassium iodide
0.02 mol dm^{-3} sodium thiosulfate
1% starch solution

Assume that all solutions are at the same temperature.

The student started the experiment by accurately measuring the solutions into a conical flask.



25.0 cm^3 of potassium iodide

10.0 cm^3 of sodium thiosulfate

1.0 cm^3 of starch solution

4.0 cm^3 of deionised water

They then added 10.0 cm^3 of 0.1 mol dm^{-3} potassium peroxydisulfate solution.

They recorded the time taken for the expected colour change to be seen.

The student needed to carry out one more run to complete the experiment.

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(i) Describe how the student should carry out the second run to complete the experiment and confirm that the reaction is first order with respect to peroxydisulfate ions.

Give the practical details, including reference to the apparatus used. State how the data would confirm that the reaction is first order with respect to peroxydisulfate ions. [6 QER]

Keep everything the same as run 1 except the concentration of $S_2O_8^{2-}$, which is halved. Using a burette (or volumetric pipettes), measure 25.0 cm^3 of potassium iodide, 10.0 cm^3 of sodium thiosulfate and 1.0 cm^3 of starch solution into a clean conical flask, with 4.0 cm^3 of deionised water as before.

Then, using a burette, measure 5.0 cm^3 of the 0.1 mol dm^{-3} potassium peroxydisulfate into a beaker and add 5.0 cm^3 of deionised water, so the total volume (50.0 cm^3) and all other concentrations stay the same as in run 1.

Add this solution rapidly to the flask, swirl to mix and start the stopwatch immediately. Stop the watch when the solution suddenly turns blue-black and record the time.

Rate is proportional to 1/time. If halving $[S_2O_8^{2-}]$ halves the rate (the time taken doubles), the reaction is first order with respect to $S_2O_8^{2-}$.

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(ii) The peroxydisulfate-iodide reaction can be catalysed by some *d*-block metal ions.

Outline how the method described in part (i) could be **modified** to investigate the catalytic effect of $Fe^{2+}(aq)$ on the reaction. State how the results would show that $Fe^{2+}(aq)$ acts as a catalyst. [2]

Repeat the experiment but replace the 4.0 cm^3 of deionised water with 4.0 cm^3 of iron(II) sulfate solution, keeping the total volume the same. If the blue-black colour forms in less time than in part (i), $Fe^{2+}(aq)$ acts as a catalyst.

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(b) Consider the cell diagram below where the negative electrode is shown on the left-hand side.

$$Pt \left| Cr^{2+}(aq), Cr^{3+}(aq) \right\| Fe^{3+}(aq), Fe^{2+}(aq) \left| Pt \right. \qquad EMF = +1.18\,V$$

(i) Give the half-equation for the reaction occurring at the negative electrode. [1]

$Cr^{2+}(aq) \rightarrow Cr^{3+}(aq) + e^-$

(ii) Give the observation at the positive electrode after the cell has been connected for a period of time. [1]

The solution becomes less yellow-brown and more green (more Fe^{2+}).

(iii) Give the overall equation for the cell reaction. Identify the oxidising agent and the reducing agent. [2]

Equation

$Cr^{2+}(aq) + Fe^{3+}(aq) \rightleftharpoons Cr^{3+}(aq) + Fe^{2+}(aq)$

Oxidising agent Fe^{3+}

Reducing agent Cr^{2+}

(iv) The concentrations of the Cr^{2+}/Cr^{3+} and Fe^{3+}/Fe^{2+} ions are all $1.0\,mol\,dm^{-3}$.

State and explain how the value on a high-resistance voltmeter would change if the concentration of the Cr^{3+} ions was decreased whilst the concentrations of all the other ions were left unchanged. [1]

Change in EMF It increases

Explanation Lowering $[Cr^{3+}]$ shifts the equilibrium $Cr^{3+} + e^- \rightleftharpoons Cr^{2+}$ to the left, so the cell equilibrium shifts to the RHS (to produce more $Cr^{3+}(aq)$) and the EMF increases.

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3. Organic acids derived from fats are described as weak acids.

(a) Explain the terms **weak** and **dilute** as applied to acid solutions. [2]

Weak: the acid only partially dissociates into its (constituent) ions when in solution.

Dilute: there is a (relatively) small amount of acid dissolved in the solution (low concentration).

(b) A student is given samples of nitric acid and ethanoic acid, both of concentration $1.0\,\text{mol}\,\text{dm}^{-3}$.

Describe **one chemical** test, apart from the use of an indicator, that could be used to distinguish between both acids.

Your answer should include an explanation of the different observations made. [2]

Add magnesium ribbon (or Na_2CO_3) to each acid. Nitric acid gives more vigorous effervescence than ethanoic acid because its $[H^+]$ is greater (it is a strong acid, fully dissociated).

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(c) A laboratory technician prepared a buffer solution of pH 5.4 by mixing 1.0 mol dm^{-3} aqueous ethanoic acid and 1.0 mol dm^{-3} aqueous sodium ethanoate.

(i) Calculate the ratio of the concentration of sodium ethanoate to ethanoic acid in the buffer mixture. [3]

(K_a for ethanoic acid = 1.78×10^{-5} mol dm^{-3} at 298 K)

$K_a = [H^+][salt] \div [acid]$

$[H^+] = 10^{-pH} = 10^{-5.4} = 3.98 \times 10^{-6}$ mol dm^{-3}

$[salt] \div [acid] = K_a \div [H^+] = 1.78 \times 10^{-5} \div 3.98 \times 10^{-6}$

ratio [salt] : [acid] = 4.47 : 1

Ratio of concentration of sodium ethanoate to ethanoic acid ..4.47.. : ..1..

(ii) Calculate the volumes of sodium ethanoate and ethanoic acid used to prepare 1 dm^3 of the buffer solution. [1]

vol salt ÷ (1000 − vol salt) = 4.47

vol salt = 4470 ÷ 5.47 = 817 cm^3; vol acid = 1000 − 817 = 183 cm^3

Volume of ethanoic acid = ..183.. cm^3

Volume of sodium ethanoate = ..817.. cm^3

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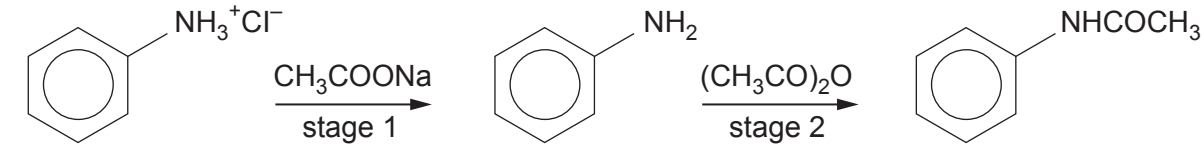
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4. Acylation is the term used to describe the substitution of a hydrogen atom in a phenol, alcohol or amine by the acyl group, RCO—.

Ethanoic anhydride, $(CH_3CO)_2O$, can be used as an acylating agent.

N-phenylethanamide, $C_6H_5NHCOCH_3$, can be prepared from the salt phenylammonium chloride, $C_6H_5NH_3^+Cl^-$. The reaction takes place in two stages.



M_r 129.6 M_r 135.1

The method below was used to prepare a pure sample of N-phenylethanamide.

- 4.78 g of phenylammonium chloride was dissolved in 150 cm^3 of deionised water in a conical flask.
- 10.0 cm^3 of ethanoic anhydride was added to the solution in the flask. The solution was stirred to ensure that all of the ethanoic anhydride had dissolved.
- 30.0 g of hydrated sodium ethanoate was dissolved in 100 cm^3 of deionised water and added to the flask. The mixture was stirred for 5 minutes.

The sodium ethanoate reacted with the phenylammonium chloride in stage 1, forming phenylamine, ethanoic acid and sodium chloride. The reaction in stage 2 happened readily with N-phenylethanamide forming as a white solid. Ethanoic acid was also formed.

The N-phenylethanamide was recrystallised to give 3.59 g of the pure product.

(a) Write the equation for the reaction occurring in stage 1. [1]

$C_6H_5NH_3Cl + CH_3COONa \rightarrow C_6H_5NH_2 + CH_3COOH + NaCl$

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(b) N-phenylethanamide is much more soluble in hot water than it is in cold water.

Use this fact to outline how you would purify N-phenylethanamide to obtain a pure, dry product. [3]

Dissolve the impure solid in the minimum volume of hot water, heating the water using a hot water bath (or hot plate).

Filter the hot solution to remove any insoluble impurities, then allow the filtrate to cool so that the N-phenylethanamide crystallises.

Filter off the crystals (under reduced pressure), wash with a small volume of cold water and dry the product.

(c) 4.78 g of phenylammonium chloride gave 3.59 g of pure N-phenylethanamide.

(i) Show that an excess of ethanoic anhydride was present in the reaction mixture. [2]

density of ethanoic anhydride = $1.08\,g\,cm^{-3}$

$n(C_6H_5NH_3Cl) = 4.78 \div 129.6 = 0.0369$ mol

mass of $(CH_3CO)_2O = 10.0 \times 1.08 = 10.8$ g

$n((CH_3CO)_2O) = 10.8 \div 102.1 = 0.106$ mol

mole ratio is 1 : 1 and $0.106 > 0.0369$, so ethanoic anhydride is in excess

(ii) Calculate the overall percentage yield of this two-stage reaction. [2]

theoretical mass of $C_6H_5NHCOCH_3 = 0.0369 \times 135.1 = 4.99$ g

percentage yield = $3.59 \div 4.99 \times 100 = 71.9$ %

Percentage yield = 71.9 %

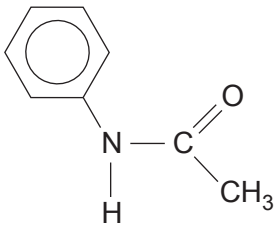
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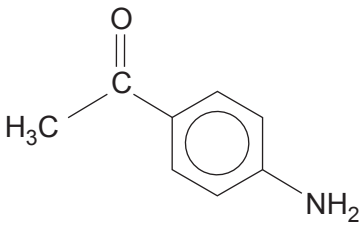
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(d) N-phenylethanamide, C_8H_9NO, and compounds **A**, **B** and **C** are structural isomers.

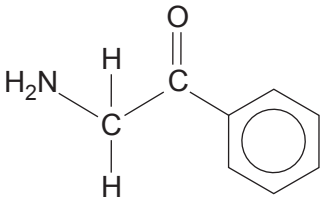
They were investigated using test tube reactions and NMR spectroscopy.



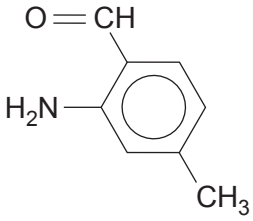
N-phenylethanamide



compound **A**



compound **B**



compound **C**

(i) Describe how the low resolution ^1H NMR spectrum of compound **C** would differ from those of the other three compounds.

Assume that all aromatic protons are equivalent. You do not need to refer to the chemical shift values or peak areas. [2]

Compound C has 4 signals (CHO, aromatic, NH_2 and CH_3 protons) in its low resolution spectrum.

The other three compounds all have 3 signals.

(ii) State a chemical test that will give a positive result for compound **C** but not for compound **A**. Give the reagent(s) and observation. [1]

Reagent(s) Tollens' reagent (ammoniacal silver nitrate), warm

Observation Silver mirror formed

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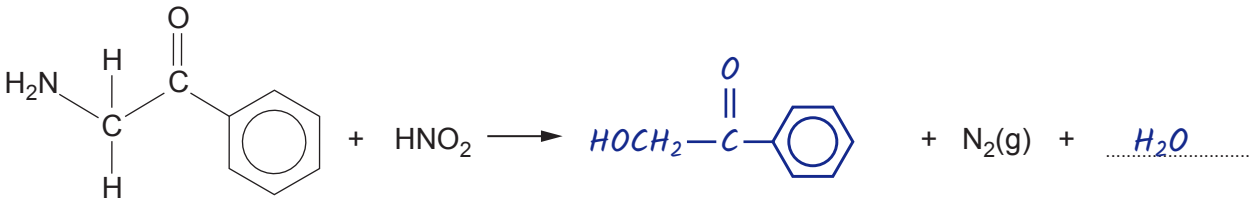
(iii) State a chemical test that will give a positive result for N-phenylethanamide and compound **A**, but not for compounds **B** and **C**. Give the reagent(s) and observation. [1]

Reagent(s) $I_2(aq)$ and NaOH(aq), warm

Observation (Pale) yellow precipitate (of CHI_3)

(iv) Nitric(III) acid (nitrous acid), HNO_2, is an unstable compound and is made when required by the reaction of a dilute acid, for example HCl, with sodium nitrate(III), $NaNO_2$.

I. Complete the equation for the reaction of compound **B** with nitric(III) acid. Give the structure of the organic compound formed. [1]



II. In an experiment, 3.59 g of compound **B** (M_r 135.1) was dissolved in water and the solution made up to 500 cm^3. A 25.0 cm^3 sample of this solution was reacted with an excess of nitric(III) acid.

Calculate the volume, in cm^3, of nitrogen gas formed at 20 °C and 1 atm. [4]

$n(B) = 3.59 \div 135.1 = 0.0266$ mol in 500 cm^3

$n(B)$ in 25.0 $cm^3 = 0.0266 \times 25.0 \div 500 = 0.00133$ mol

$n(N_2) = n(B) = 0.00133$ mol

$V = nRT \div p = (0.00133 \times 8.31 \times 293) \div 1.01 \times 10^5$

$V = 3.21 \times 10^{-5}\ m^3 = 32.1\ cm^3$

Volume = 32.1 cm^3

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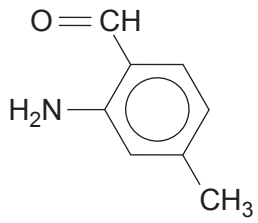
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(v) Compound **D** can be prepared from compound **C** in a two-stage process.

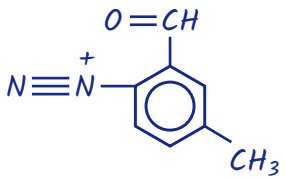
Give the structures of the intermediate and compound **D**. [2]

compound **C**



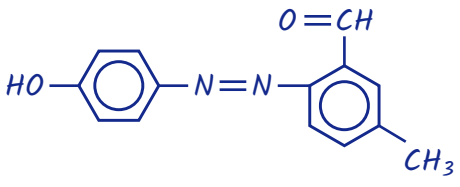
HNO_2
temperature
below 10°C

intermediate



alkaline phenol
temperature
below 10°C

compound **D**



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5. Tutton's salts are a family of double salts with the formula $A_wB_x(SO_4)_y.z(H_2O)$. They contain two different cations, A^{a+} and B^{b+}.

The following tests were carried out on a Tutton salt where A^{a+} is a Group 1 cation and B^{b+} is a transition metal cation. A solution of the salt was prepared for use in tests 2 and 3.

Complete the table. [12]

Test	Result	Conclusion
Test 1 Heat the salt to constant mass	0.05 mol sample of the salt loses 5.41 g of water	$n(H_2O) = 5.41 \div 18.02 = 0.30$ mol mole ratio salt : $H_2O = 0.05 : 0.30$ $= 1 : 6$, so $z = 6$ $z =$ 6

Test	Result	Conclusion
Test 2A Add NaOH(aq) dropwise and then in excess (to the solution)	(dark) green precipitate, insoluble in excess NaOH	Cr^{3+} not present so B^{b+} is Fe^{2+}
Equation $Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)$		
Test 2B Leave the precipitate that forms exposed to the air for 24 hours	Precipitate turns reddish-brown	$Fe(OH)_2$ (Fe^{2+}) is oxidised by the air to $Fe(OH)_3$ (Fe^{3+}), which is reddish-brown

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Test	Result	Conclusion
Test 3A Add $BaCl_2(aq)$ (acidified with dilute HCl)	(heavy) white precipitate forms	SO_4^{2-} present
Equation $Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$		
Test 3B Add excess reagent	50.0 cm^3 sample of a 0.736 mol dm^{-3} solution of the salt produces a precipitate with a dry mass of 17.16 g	precipitate is $BaSO_4$ (M_r 233.1) $n(BaSO_4) = 17.16 \div 233.1 = 0.0736$ mol $n(salt) = 0.736 \times 50.0 \div 1000 = 0.0368$ mol mole ratio salt : SO_4^{2-} = 0.0368 : 0.0736 = 1 : 2, so $y = 2$ $y =$ 2

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Test	Result	Conclusion
Test 4 Elemental analysis	Hydrated salt (M_r 434.3) contains 12.85% by mass of B^{b+}	B^{b+} is Fe^{2+} (A_r 55.8) mass of B = 12.85 ÷ 100 × 434.3 = 55.8 so one Fe per formula, x = 1 $Fe(SO_4)_2.6H_2O$ = 55.8 + 192.2 + 108.1 = 356.1 mass of A = 434.3 − 356.1 = 78.2 78.2 = 2 × 39.1: A is K^+, w = 2 x = 1 w = 2

Formula of Tutton salt is $K_2Fe(SO_4)_2.6H_2O$

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END OF PAPER

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Question number	Additional page, if required. Write the question number(s) in the left-hand margin.

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