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CANDIDATE NAME	
CLASS	2T

CHEMISTRY

9729/04

Paper 4 Practical

23 August 2022 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 20 and 21.

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.

ANSWERS for review

Shift Laboratory

For Examiner's Use	
1	/ 15
2	/ 13
3	/ 27
Total	/ 55

This document consists of **24** printed pages and **0** blank page.

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

1 Determination of the M_r of a hydrated ethanedioate salt, using acidified potassium manganate(VII) by titration

Calcium ethanedioate is the major component of the most common type of human kidney stones. It is one of a series of salts formed from ethanedioic acid, $H_2C_2O_4$. Another of these salts can be represented by the formula $X_2C_2O_4 \cdot H_2O$, where X is a Group 1 metal.

Solution **Q** contains 64.5 g dm⁻³ of **X**₂C₂O₄•H₂O in deionised water. You are not provided with **Q**.

FA 1 is a diluted solution of \mathbf{Q} , in which 35.70 cm³ of \mathbf{Q} was made up to 250 cm³ with deionised water in a graduated flask.

FA 2 is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄. **FA 3** is 1.00 mol dm⁻³ sulfuric acid, H₂SO₄.

In this question, you will perform a titration. The data from this titration will be used to determine:

- the concentration of $C_2O_4^{2-}$ ions in **Q**.
- the M_r of $X_2C_2O_4$ •H₂O, and hence the identity of the metal X.

(a) Titration of FA 1 against FA 2

In this titration, **FA 2** is run from a burette into the conical flask containing **FA 1** and **FA 3**. Initially, the colour of the **FA 2** will take some time to disappear. After some **FA 2** has been added, sufficient $Mn^{2+}(aq)$ ions will be present to allow the reaction to occur faster.

The end point is reached when a permanent **pale** pink colour is obtained.

- (i) 1. Fill the burette with **FA 2**.
 - 2. Using a pipette, transfer 25.0 cm³ of **FA 1** into the conical flask.
 - 3. Using an appropriate measuring cylinder, transfer 25.0 cm³ of **FA 3** to the same conical flask.
 - 4. Heat this solution to about 60 °C.
 - 5. Run **FA 2** from the burette into this flask until a permanent **pale** pink colour is obtained. Be careful when you titrate under the hot condition.
 - 6. Record your titration results to an appropriate level of precision, in the space provided next page.
 - 7. Repeat points **1** to **6** until consistent results are obtained.
 - 8. Turn off your Bunsen burner.



Titration results

	1	2
Final burette reading /cm ³	24.95	49.90
Initial burette reading /cm ³	0.00	24.95
Volume of FA 2 used /cm ³	24.95	24.95

- Tabulate initial and final burette readings and volume used in the titration table and table has correct headers and units.
- Consistent burette readings taken (V_{FA 2} ± 0.10 cm³) and recorded to 2 d.p.
- Accuracy of titration results.

[5]

(ii) From your titrations, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

Average volume of FA 2 = $(\frac{24.95+24.95}{2}) = 24.95 \text{ cm}^3$

volume of **FA 2** =[1]

(iii) The equation for the reaction between ethanedioate ions, $C_2O_4^{2-}$ and manganate(VII) ions is shown below.

$$5C_2O_4^{2-}(aq) + 2MnO_4^{-}(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 8H_2O(l) + 10CO_2(g)$$

Calculate the amount of ethanedioate ions, $C_2O_4^{2-}$ in 25.0 cm³ of FA 1. Amount of $MnO_4^- = (\frac{24.95}{1000} \times 0.0200) = 4.99 \times 10^{-4}$ mol

[Turn over

(iv) Determine the concentration in mol dm⁻³, of $C_2O_4^{2-}$ in Q. (concentrated solution) Amount of $C_2O_4^{2-}$ in 250 cm³ of FA 1 = $(\frac{250}{25.0} \times 1.25 \times 10^{-3})$ mol = $(10 \times 1.25 \times 10^{-3})$ mol = 0.0125 mol Amount of $C_2O_4^{2-}$ present in 35.70 cm³ of Q = 0.0125 mol Concentration of $C_2O_4^{2-}$ in Q = $(\frac{1000}{35.70} \times 0.0125)$ mol

 $= 0.349 \text{ mol dm}^{-3}$

(v) Use your answer to (a)(iv) to calculate the M_r of the ethanedioate salt. Given: solution Q contains 64.5 g dm⁻³ of X₂C₂O₄•H₂O. M_r of X₂C₂O₄•H₂O = $\frac{64.5}{0.349}$ = 184.8

 M_r of the ethanedioate salt =[1]

(vi) Hence, deduce the identity of X. Show your working clearly.
[A_r: C, 12.0; O, 16.0; H, 1.0; Li, 6.9; Na, 23.0; K, 39.1; Rb, 85.5; Cs, 132.9; Fr, 223.0]
M_r of X₂C₂O₄●H₂O = 184.8
2(A_r of X) + 88.0 + 18.0 = 184.8
A_r of X = 39.4
So, X is K or potassium

K or potassium X is[2]

(b) A student performed the experiment in (a)(i) using a sample of another ethanedioate salt. The student obtained a mean titre value of 22.20 cm³.

The teacher calculated that the volume of **FA 2** required should be 22.40 cm³. The teacher told the student that the total percentage error from the apparatus in the experiment was 0.4%.

Calculate the error in the student's result, based on these data. State and explain whether or not the student's result is accurate.

Experimental error = $(\frac{22.40-22.20}{22.40})$ x 100% = 0.893% Candidate states that experiment is not accurate and justifies this in terms of experimental error being > apparatus error. [2] [7] [Total: 15] [Turn over

2 Investigation of reaction between manganate(VII) ions and ethanedioate ions

FA 2 is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄ (same as in Q1).

- **FA 3** is 1.0 mol dm⁻³ sulfuric acid, H_2SO_4 (same as in Q1).
- $\label{eq:FA4} \textbf{FA4} \quad \text{is 0.200 mol } dm^{-3} \text{ ethanedioic acid, } H_2 C_2 O_4.$
- **FA 5** is 0.0100 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃.
- **FA 6** is 0.100 mol dm⁻³ potassium iodide, KI.

You are also provided with a starch indicator.

Acidified potassium manganate(VII) oxidises ethanedioate ions, $C_2O_4^{2-}$ as shown in **reaction 1**. The Mn²⁺ ions produced in **reaction 1** act as a catalyst for the reaction. This is an example of 'autocatalysis'.

$$2MnO_4^{-}(aq) + 5C_2O_4^{2-}(aq) + 16H^{+}(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$
 reaction 1

You are to investigate how the rate of reaction changes with $[MnO_4^-]$. To do this, you will prepare a reaction mixture containing **FA 2**, **FA 3** and **FA 4**. At timed intervals, you will withdraw **five aliquots** (portions) of the reaction mixture, add them to 10 cm³ of excess KI which will "quench" the reaction by reacting away all MnO_4^- via a redox reaction as shown in **reaction 2**.

$$2MnO_4^{-}(aq) + 10I^{-}(aq) + 16H^{+}(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(l)$$
 reaction 2

You will then titrate the iodine produced in the resulting solutions against sodium thiosulfate (**reaction 3**).

$$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow 2I^{-}(aq) + S_4O_6^{2-}(aq)$$
 reaction 3

Your titre values will indicate the concentration of MnO_4^- remaining in the reaction mixture at different times. Hence, the rate of reaction between MnO_4^- and $C_2O_4^{2-}$ at different times can be determined by graphical analysis of your results.

Amount of MnO_4^- present at that instantaneous time can be found by finding the amount of iodine that has reacted with thiosulfate.

1. At the start, before the reaction

		MnO₄ [−]	
2.	1:00 minute		
	N	InO₄ [−]	
3.	4:00 minute		
	MnO₄ [−]		
4.	7:00 minute		
	MnO₄⁻		
5.	10:00 minute		
	MnO ₄ -		
6.	14:00 minute		
ſ	√InO₄ [−]		

You should read all of the instructions on this page and the method on the next page before you start this experiment.

Recording your results

In an appropriate format in the space provided on **page 7** under the heading, **Results**, in **(b)**, prepare a table to record, for each of your aliquots, the

- transfer time in minutes and seconds,
- titration results (initial and final burette readings; and volume of FA 5 added),
- time, t, which is the transfer time converted to minutes, to one decimal place (e.g. a transfer time of 2 min 27 s becomes 2 min + 27/60 min = 2.5 min).

Make certain that your recorded results show the precision of your working.

(a) Method

Preparing the boiling tubes and burette

- 1. Using a 10 cm³ measuring cylinder, add about 10 cm³ of **FA 6** to each of the labelled boiling tubes, **1** to **5**.
- 2. Fill a burette with FA 5.

Preparing the reaction mixture

- 3. Use appropriate measuring cylinders to add to the beaker labelled **reaction mixture**
 - 5.0 cm³ of **FA 3**,
 - 50.0 cm³ of **FA 4**,
 - 45.0 cm³ of deionised water.

- 4. Place 25.0 cm³ of **FA 2** into a 25 cm³ measuring cylinder.
- 5. At a convenient time, pour **FA 2** into the beaker labelled **reaction mixture**. Start the stopwatch at the instant of mixing and stir the mixture thoroughly using a glass rod.
- **Note:** Once you have started the stopwatch, allow it to continue running for the duration of the experiment. You **must not stop** the stopwatch until you have collected all of your aliquots.

Removing aliquots of reaction mixture

- **Note:** Leaving all of the titrations to be performed until after all the aliquots have been collected may cause you time problems.
- 6. At approximately 1 minute, use a 10 cm³ pipette to remove a 10.0 cm³ aliquot of the reaction mixture. Immediately transfer this aliquot into the boiling tube labelled 1 and shake the mixture. Note and record the transfer time (in minutes and seconds, to the nearest second) when half of the reaction mixture has emptied from the pipette.
- 7. At approximately 4 minutes, repeat point **6**. Transfer this aliquot into the boiling tube labelled **2** and shake the mixture.
- 8. Repeat point **6** three more times at about 7 minutes, 10 minutes and 13 minutes, transferring the aliquots into the boiling tubes labelled **3** to **5**.

Titrations

- **Note:** Each titration is to be performed **once only**, so great care should be taken that you do not overshoot the end-point.
- 9. Pour all the contents of boiling tube **1** into a clean conical flask. Rinse this boiling tube with deionised water and add the washings to the conical flask.
- 10. Record the initial burette reading. Titrate the liberated I₂ in this solution with FA 5 until the solution turns pale yellow. Then add about 1 cm³ of starch indicator. The solution will turn blue-black. Continue to titrate until the blue-black colour just disappears at the end-point. Record the final burette reading and the volume of FA 5 added.
- 11. Wash this conical flask thoroughly with water.
- 12. Top up the burette with FA 5.
- 13. Repeat points 9 to 11 as required for each of the remaining boiling tubes.

(b) Results

Transfer time	Final burette reading /cm ³	Initial burette reading /cm ³	Volume of FA 5 added /cm ³	t /min
1 min 20 s	19.00	0.00	<mark>19.00</mark>	<mark>1.3</mark>
4 min 25 s	17.70	0.00	<mark>17.70</mark>	<mark>4.4</mark>
7 min 25 s	30.60	17.70	<mark>12.90</mark>	<mark>7.4</mark>
10 min 12 s	35.60	30.60	<mark>5.00</mark>	<mark>10.2</mark>
13 min 20 s	37.40	35.60	<mark>1.80</mark>	<mark>13.3</mark>

[5]

- Tabulates transfer time, initial and final burette readings, volume of FA 5 added and t in a single table with correct headers and correct units.
- Records all 5 full sets transfer times in whole no. in min and s and converts all values of t correctly to 1 decimal place in min.
- Records all 5 full sets of initial and final burette readings + calculates all titres correctly to 2 d.p.
- Enters all 5 full sets of data with all transfer times within range of suggested times.
- Titre for first aliquot within range + decreasing titre with increasing time, t.

(c) (i) On the grid below, plot a graph of the volume of sodium thiosulfate, **FA 5**, on the y-axis, against time, t, on the x-axis.

Draw the most appropriate curve taking into account all of your plotted points.



- Axes correct way round + axes with correct labels with units + uses proper scale (not awkward scale) with plotted points occupying at least half of the given grid in both directions.
- All points plotted correctly to within $\pm \frac{1}{2}$ small square.
- A smooth curve (correct shape) of best-fit drawn, based on student's results.

(ii) The rate of reaction at time t is indicated by the slope of the tangent at time t.

Describe and explain how the **shape** of the graph in (c)(i) shows that the reaction between MnO_4^- and $C_2O_4^{2-}$ is an autocatalysed reaction.

At the start, the slope of the tangent is <u>almost zero/small/gentle</u>. This indicates that the <u>rate of reaction is low</u> due to absence of $Mn^{2+}/catalyst$ or low concentration of $Mn^{2+}/catalyst$.

As the reaction proceeds, the <u>slope/gradient of the tangent increases</u>. This indicates that the <u>rate of reaction increases</u> due to more catalyst/ Mn^{2+} is produced or concentration of Mn^{2+} /catalyst increases.

Towards the end, the slope of the tangent decreases. This indicates that the rate of reaction decreases due to decrease in concentration of reactants. A student performed a similar experiment in cooler conditions. In point 3, she used the same volumes of **FA 3** and **FA 4** that you used but she also added 5.0 cm³ of a solution of manganese(II) sulfate, $MnSO_4$, a source of Mn^{2+} to catalyse the reaction. She only added 40.0 cm³ of deionised water, so the total volume used was the same as in your experiment.

(d) On the grid below, the data from the student's experiment has been plotted and the graph line has been drawn.



[Turn over

Use data from the graph in (d) to determine the order of reaction with respect to $[MnO_4^-]$ in **reaction 1**. Draw clearly any construction lines on the graph. Explain your reasoning clearly.

Volume of $S_2O_3^{2-}$ (FA 5) required at time t is directly proportional to the [MnO₄⁻] left in the reaction mixture at time t.

From the graph, first
$$t_{1/2} = 13.00$$
 min,
second $t_{1/2} = 13.00$ min,
third $t_{1/2} = 13.50$ min.

Indicate the $t_{1/2}$ values correctly (based on working) either on the graph or in this space.

Shows clear construction lines on the graph to determine the $t_{1/2}$ values (showing the initial V for each $t_{1/2}$ clearly).

Since half-lives are (approximately) constant, the reaction is first order with respect to $[MnO_4^-]$ in reaction 1.

[3] [Total: 13]

3 Part 1: Qualitative Analysis

In this task you are to explore the chemistry of some compounds of an unknown transition element \mathbf{R} and determine the identities or structures of a number of different substances.

- **FB1** is a solid sample of a common dioxide of the unknown transition element **R**.
- **FB 2** is dilute sulfuric acid, H₂SO₄.
- **FB 3** is a solid sample of sodium ethanedioate, $Na_2C_2O_4$.
- FB 4 is a solution of pure compound S, which is the product that will be formed in (a)(i).
- FB 5 is a dilute solution of KMnO_{4.}
- **FB 6** is a simple hydrocarbon compound.
- **FB7** is bromine water.

Carry out the following experiments. Carefully record your observations in the spaces provided. Test any gases produced.

		Test	Observations
	(a) (i)	Transfer all of the solid sample of	1. White solid (Na ₂ C ₂ O ₄) eventually
solic	I Na ₂ C ₂ O ₄	 FB 3 into a small conical flask. Add 25 cm³ of FB 2 to the flask. dilute sulfuric acid 	dissolves during the experiment.
		Gently heat the flask until the temperature of the mixture reaches about 60 °C.	
		Swirl the mixture carefully. Place the flask on the wire gauze / heat proof mat.	
		RO ₂ Using a spatula, add FB 1 to the mixture in small portions. Between each addition, stir the mixture carefully with the thermometer and	 2. Effervescence/bubbles after each addition (do not accept "gas evolved"). 3. CO₂ gas (collected) gives a white
		temperature of the mixture.	ppt with lime water.
		80.	4. Upon addition of FB1, temperature of mixture rises / Conical flask feels hot (even though no heat is applied).
		Stop adding FB 1 to the mixture when you think the reaction is complete.	5. Temperature rise stops /temperature falls / mixture starts to cool / effervescence much less vigorous on addition of more FB 1.

Filter the mixture into a boiling tube and leave the filtrate to stand. The filtrate contains the compound S .	6. (Pale pink) filtrate turns yellow- green /pale yellow.
Retain this filtrate for use in (a)(ii) .	

(ii) In this part, you are to investigate the effect of the addition of aqueous sodium hydroxide, and the addition of ammonia, to separate portions of the filtrate from (a)(i) and FB 4.

solution of compound S

In the space below, prepare a suitable table and in it record details of the tests performed and the observations made.

	Test	Observations
(a)(ii)	Place 2 cm ³ of the filtrate from (a)(i) in a test-tube, add to it dropwise NaOH(aq), with shaking, till excess.	 Tests with the filtrate from (a)(i): 7. NaOH(aq) and NH₃(aq): off -white / light brown ppt.
	To 2 cm ³ of the filtrate from (a)(i) in another test-tube add to it dropwise $NH_3(aq)$, with shaking, till excess.	Tests with FB 4: 8. NaOH(aq) and NH ₃ (aq): off-white / light brown ppt.
	Repeat the above tests using NaOH(aq) and NH ₃ (aq) separately with FB 4 , in place of the filtrate from (a)(i) . solution of compound	9. NaOH(aq) and NH ₃ (aq) with both solutions: all 4 ppts insoluble in excess.
	A single table to describe the procedure of adding dropwise / small volume of NaOH(aq) and NH ₃ (aq) and then in excess, both with shaking, to separate samples of the filtrate from (a)(i) and FB 4.	10. NaOH(aq) and NH₃(aq) with both solutions: ppts darken (on standing) / turns brown on contact with air

	Test	Observations	
(b) (i)	Place 5 cm ³ of aqueous sodium hydroxide in a test-tube.		
	simple hydrocarbon Add 1 drop of FB 6 to this test-tube.		
	dilute solution of KMnO ₄ Add FB 5 , dropwise with shaking,	Solution turns green / blue-green.	
	not exceed 40 drops.	Colour deepens as more drops are added.	
	Note: Eventually, this reaction will pro-	duce a precipitate of FB 1 .	
(b) (ii)	Place 5 cm ³ of FB 2 in a test-tube. H₂SO₄(aq)		
	Add 1 drop of FB 6 to this test-tube. simple hydrocarbon		
	Add FB 5 , dropwise with shaking, until no further change is seen. Do	Purple FB 5 decolourises.	
	not exceed 40 drops.	Colourless solution turns	
		progressively darker brown.	
(b) (iii)	Place 5 cm ³ of deionised water in a test-tube.		
	simple hydrocarbon Add 1 drop of FB 6 to this test-tube.		
	bromine water Add FB 7 , dropwise with shaking, until no further change is seen.	Orange bromine water decolourised (solution eventually turns yellow).	
		[9]	

Note that the observations for tests (b) are provided as shown below.

Conclusions

RO₂ solid Na₂C₂O₄

(c) (i) In (a)(i), the reaction between **FB 1** and **FB 3** occurs under acidic conditions.

Write an ionic equation for this reaction. Use RO_2 to represent FB 1 in this equation.

(ii) Consider your observations in (a)(ii).
 Identify the transition metal ion formed in (a)(i). Justify your choice by reference to your observations in (a)(ii).

	ion present is ^{Mn²+}
	justification
	Off-white.ppt.formed.with.both.NaOH.and.NH2.which.are.insoluble.in
	excess. (and which turns.darker.on standing in air)[2]
(d)	dilute solution of KMnO₄green/ blue-greenConsider the colour of compound FB 5 and the observations provided in (b)(i).Suggest a value for the oxidation number of R in the coloured ion produced in (b)(i).Explain your reasoning.
	oxidation number of $R =$
	explanation .The oxidation numbers of R./.Mn in compound, FB 5 and in FB 1 are +7 and +4 respectively.
	So, as the reaction in (b)(i) involves a change of O.N. from +7 to +4, the O.N.
	of the coloured ion will be in between these values.
	[2]

Some Oxidation States of Manganese



*Most common states in bold face.

- (e) Compound U is the main organic product in (b)(i), when FB 6 reacts with FB 5 under alkaline conditions. The molecular formula of U is C₆H₁₂O₂.
 difference of 20 and 2H
 - Deduce the molecular formula of FB 6. (i) simple hydrocarbon Explain your deduction. Your explanation should be supported by evidence from the observations provided in (b). molecular formula of **FB 6** is ...C₆H₁₀ Br₂(aq) decolourised, so C=C present / FB 5 or KMnO₄ explanation ... decolourised so C=C present. Cold alkaline MnO₄⁻ oxidises C=C to C(OH)-C(OH) / 1,2-diol. OR Mild oxidation Only two O atoms in molecular formula of U, so only one C=C. • So, FB 6 is $C_6H_{12}O_2 - (2 \times OH) = C_6H_{10}$. [2] simple hydrocarbon (ii) Draw the structural formulae for FB 6 and compound U. structure of FB 6 structure of U





[2]

Part 2: Planning

(a) Four liquid samples labelled FC 1, FC 2, FC 3 and FC 4 are listed below.



The identity of each of the liquid samples is not known. A student carried out a series of test-tube reactions so as to distinguish the four liquid samples. The tests he performed and the corresponding observations are recorded in the table below.

	Test	Observations	Observations	Observations	Observations
(1)		with FC 1	with FC 2	with FC 3	with FC 4
(1)	 I o 1 cm depth of the sample in a test-tube, add about 1 cm³ of aqueous bromine. 	Orange aqueous bromine is decolourised.	No observable change.	No observable change.	No observable change.
	 To 1 cm depth of the sample in a test-tube, add 10 drops of aqueous H₂SO₄ and 5 drops of aqueous KMnO₄. Place the test-tube in a hot water bath. 	Purple acidified KMnO ₄ is decolourised.	Purple acidified KMnO4 is decolourised.	Purple acidified KMnO4 is decolourised.	No observable change.
	 To 1 cm depth of the sample in a test-tube, add 6 drops of aqueous NaOH, followed by 10 drops of aqueous I₂. Place the test-tube in a hot water bath. 	No observable change.	A yellow ppt of CHI ₃ is seen.	No observable change.	A yellow ppt of CHI ₃ is seen.

(ii) Now, based on the student's tests and observations, identify the four liquid samples, FC 1, FC 2, FC 3 and FC 4.

In each case, give evidence to support your conclusion, by completing the table below.

	liquid sample	evidence
FC 1	Cyclohexene.	It <u>decolourises</u> orange <u>Br₂(aq)</u> , indicating the presence of <u>C=C or alkene</u> . It also <u>decolourises</u> <u>acidified KMnO₄(aq)</u> as it undergoes oxidative cleavage.
FC 2	Propan-2-ol.	It <u>decolourises</u> purple acidified <u>KMnO₄(aq)</u> as a <u>secondary alcohol</u> is oxidised. It also gives a <u>positive iodoform reaction</u> due to the presence of the group, <u>CH₃CH(OH)–</u> .
FC 3	Benzaldehyde.	It <u>decolourises purple acidified KMnO₄(aq)</u> as an <u>aldehyde</u> is oxidised.
FC 4	Propanone.	It gives a <u>positive iodoform reaction</u> as it is a ketone with the presence of the group, <u>-COCH₃</u>

[4]

(b) Consider the following organic compounds.



Plan an investigation, using test-tube reactions, **other** than those used in **(a)** which would enable you to identify each of these four compounds.

Each compound should be identified by **only one** positive test result. It is not sufficient to identify simply by eliminating all the others.

Your plan should include:

- details of the reagents and conditions to be used,
- an outline of the sequence of steps you would follow,
- an explanation of how you would analyse your results in order to identify each compound.

Once a compound has been clearly identified, your plan should concentrate on distinguishing the remaining compounds.

YOU MUST NOT CARRY OUT THIS PLAN.

- 1. Add <u>Tollens' reagent to the 4 compounds separately in a test tube. Heat gently in a water bath. Butanal</u>, an aldehyde will give a <u>silver mirror</u>, the <u>other 3 compounds will</u> not give a silver mirror.
-(Accept Fehling's solution with the corresponding observation.).....
- 2. Add 2,4-DNPH to the remaining 3 compounds separately in a test-tube. Heat gentlyin a water bath. Propanone, a ketone will give an orange ppt, the other 2 compounds will not give an orange ppt.
- 3...Add <u>K₂Cr₂O₇(aq) and H₂SO₄(aq)</u> to the remaining 2 compounds separately in a testtube. <u>Heat</u> gently in a water bath. <u>Propan-1-ol</u> being a primary alcohol, is oxidised and the <u>orange</u> acidified K₂Cr₂O₇ turns green.
-<u>2-methylpropan-2-ol</u>, being a tertiary alcohol is not oxidised by acidified $K_2Cr_2O_7$. So, the orange acidified $K_2Cr_2O_7$ does not turn green.
- [Do not accept KMnO4(aq) and H2SO4(aq) used in (a)]

4. Add anhydrous <u>PCI₅</u> to the remaining compound in a test-tube. Steamy <u>white fumes</u>

of HC/ are observed, indicating that it is an alcohol, 2-methylpropan-2-ol.

(Accord No model COC) with the compared in a charactions)

(Accept Na metal, SOCl₂ with the corresponding obervations.)

Correct sequence of steps used:

- 2,4-DNPH is not used as the first step as it gives a positive test with both butanal and propanone. [Total: 27]

- acidified K₂Cr₂O₇(aq) is not used as the first step as both butanal and propan-1ol can both be oxidised.
- PCl₅, Na metal and SOCl₂ are not used as the first step as each gives a positive test with both the alcohols [Turn over

Qualitative Analysis Notes [ppt. = precipitate]

(a) Reactions of aqueous cations

action	reaction with		
Callon	NaOH(aq)	NH₃(aq)	
aluminium, Al ³⁺ (aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH₄⁺(aq)	ammonia produced on heating	_	
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.	
calcium, Ca²+(aq)	white ppt. with high [Ca ²⁺ (aq)]	no ppt.	
chromium(III), Cr³⁺(aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess	
copper(II), Cu ²⁺ (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution	
iron(II), Fe ²⁺ (aq)	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess	
iron(III), Fe ³⁺ (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess	
magnesium, Mg²+(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	

ion reaction carbonate, CO₂ liberated by dilute acids CO32chloride, gives white ppt. with $Ag^+(aq)$ (soluble in $NH_3(aq)$) Cl⁻(aq) bromide, gives pale cream ppt. with $Ag^+(aq)$ (partially soluble in $NH_3(aq)$) Br⁻(aq) iodide, gives yellow ppt. with $Ag^{+}(aq)$ (insoluble in $NH_{3}(aq)$) I⁻(aq) nitrate, NH₃ liberated on heating with OH⁻(aq) and Al foil NO₃⁻(aq) NH_3 liberated on heating with $OH^-(aq)$ and Al foil; nitrite, NO liberated by dilute acids NO₂⁻(aq) (colourless NO \rightarrow (pale) brown NO₂ in air) sulfate, gives white ppt. with Ba²⁺(aq) (insoluble in excess dilute strong acids) SO42-(aq) SO₂ liberated with dilute acids; sulfite, gives white ppt. with Ba²⁺(aq) (soluble in dilute strong acids) $SO_3^{2-}(aq)$

(b) Reactions of aqueous anions

(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, I2	black solid / purple gas	brown	purple