Catholic Junior College

JC2 Preliminary Examinations
Higher 2

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 19 and 20.

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	**/ 15**
2	**/ 13**
3	**/ 27**
Total	**/ 55**

This document consists of **20** printed pages.

[Turn over

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 $\mathbf{X}_2C_2O_4{\bullet}H_2O$, where **X** is a Group 1 metal.

Solution **Q** contains 64.5 g dm^{-3} of $\mathbf{X}_2C_2O_4{\bullet}H_2O$ in deionised water. You are not provided with **Q**.

FA 1 is a diluted solution of **Q**, in which 35.70 cm^3 of **Q** was made up to 250 cm^3 with deionised water in a graduated flask.
FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.

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 $\mathbf{X}_2C_2O_4{\bullet}H_2O$, 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 cm^3 of **FA 1** into the conical flask.
3. Using an appropriate measuring cylinder, transfer 25.0 cm^3 of **FA 3** to the same conical flask.
4. Heat this solution to about 60 ^{o}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

[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.

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 cm^3 of **FA 1**.

amount of $C_2O_4^{2-}$ in in 25.0 cm^3 of **FA 1** = …………………………..[2]

(iv) Determine the concentration in mol dm^{-3}, of $C_2O_4^{2-}$ in **Q**.

concentration of $C_2O_4^{2-}$ in **Q** = …………………………..[2]

[Turn over

(v) Use your answer to **(a)(iv)** to calculate the M_r of the ethanedioate salt.

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]

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 cm^3.

The teacher calculated that the volume of **FA 2** required should be 22.40 cm^3. 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**.**

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[Total: 15]

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

FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$ **(same as in Q1)**.
FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 **(same as in Q1)** .
FA 4 is 0.200 mol dm^{-3} ethanedioic acid, $H_2C_2O_4$.
FA 5 is 0.0100 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
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 Mn^{2+} 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 cm^3 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.

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.

[Turn over

(a) Method

Preparing the boiling tubes and burette

1. Using a 10 cm^3 measuring cylinder, add about 10 cm^3 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 cm^3 of **FA 3**,
   - 50.0 cm^3 of **FA 4**,
   - 45.0 cm^3 of deionised water.

4. Place 25.0 cm^3 of **FA 2** into a 25 cm^3 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 cm^3 pipette to remove a 10.0 cm^3 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 I_2 in this solution with **FA 5** until the solution turns pale yellow. Then add about 1 cm^3 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

[5]

(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.

[3]

(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.

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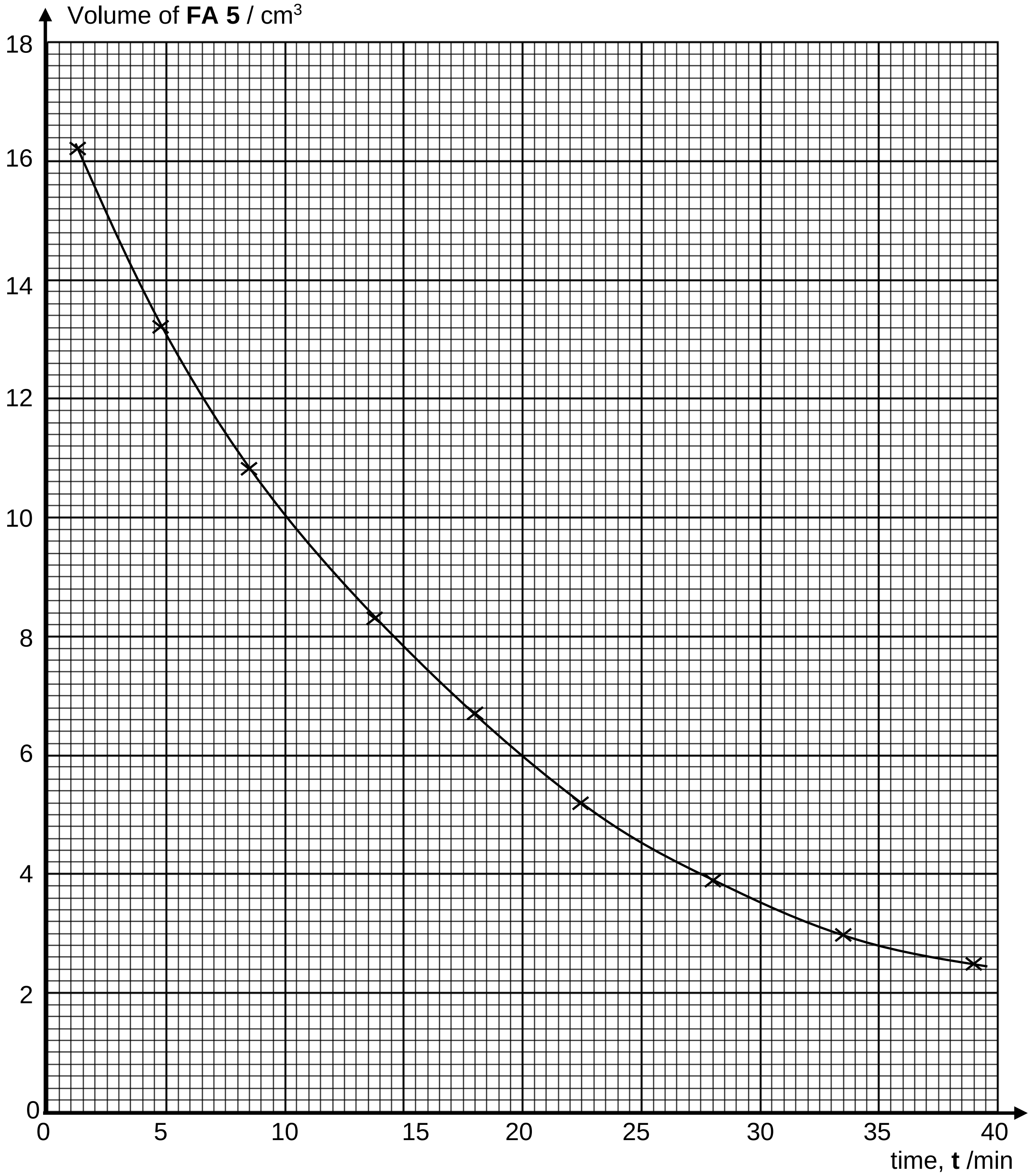
....[2]

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 cm^3 of a solution of manganese(II) sulfate, $MnSO_4$, a source of Mn^{2+} to catalyse the reaction. She only added 40.0 cm^3 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.

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[Total: 13]

3 Part 1: Qualitative Analysis

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

FB 1 is a solid sample of a common dioxide of the unknown transition element **R**.
FB 2 is dilute sulfuric acid, H_2SO_4.
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.
FB 7 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 **FB 3** into a small conical flask. Add 25 cm^3 of **FB 2** to the flask. Gently heat the flask until the temperature of the mixture reaches about 60 ^oC. Swirl the mixture carefully. Place the flask on the wire gauze / heat proof mat. Using a spatula, add **FB 1** to the mixture in small portions. Between each addition, stir the mixture carefully with the thermometer and observe any changes in the temperature of the mixture. Stop adding **FB 1** to the mixture when you think the reaction is complete. Filter the mixture into a boiling tube and leave the filtrate to stand. The filtrate contains the compound **S**. Retain this filtrate for use in **(a)(ii)**.	

[Turn over

(a) (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**.

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

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

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

[9]

[Turn over

Conclusions

(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 **R**O_2 to represent **FB 1** in this equation.

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(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 ……………………………………………………………………..........

justification ……………………………………………………………………………….

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(d) Consider 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 ……………………………………………………………………………...

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(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 $C_6H_{12}O_2$.

(i) Deduce the molecular formula of **FB 6**.

Explain your deduction. Your explanation should be supported by evidence from the observations provided in **(b)**.

molecular formula of **FB 6** is ………………………………………………………….

explanation ……………………………………………………………………………

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....[2]

(ii) Draw the structural formulae for **FB 6** and compound **U**.

[2]

[Turn over

Part 2: Planning

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

benzaldehyde　　cyclohexene　　propan-2-ol　　propanone

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 with **FC 1**	Observations with **FC 2**	Observations with **FC 3**	Observations with **FC 4**
(i)	1. To 1 cm depth of the sample in a test-tube, add about 1 cm^3 of aqueous bromine.	Orange aqueous bromine is decolourised.	No observable change.	No observable change.	No observable change.
	2. To 1 cm depth of the sample in a test-tube, add 10 drops of aqueous H_2SO_4 and 5 drops of aqueous $KMnO_4$. Place the test-tube in a hot water bath.	Purple acidified $KMnO_4$ is decolourised.	Purple acidified $KMnO_4$ is decolourised.	Purple acidified $KMnO_4$ is decolourised.	No observable change.
	3. To 1 cm depth of the sample in a test-tube, add 6 drops of aqueous NaOH, followed by 10 drops of aqueous I_2. 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		
FC 2		
FC 3		
FC 4		

[4]

[Turn over

(b) Consider the following organic compounds.

butanal   2-methylpropan-2-ol   propan-1-ol   propanone

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.

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[Total: 27]

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, 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 A*l* foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and A*l* foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (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 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