CANDIDATE NAME	( )	CLASS	20S0		

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RAFFLES INSTITUTION
2020 YEAR 6 PRELIMINARY EXAMINATION

Higher 2

CHEMISTRY 9729/04

Paper 4 Practical

26 August 2020

2 hours 30 minutes

Do not turn over the Question Booklet until you are told to do so.

READ THESE INSTRUCTIONS FIRST.

Write your name and class on the space provided when instructed to do so.
Give details of the practical shift and laboratory where appropriate, in the space 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 number of marks is given in brackets [ ] at the end of each question or part question.

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.

Shift	
Laboratory	
Bench Number	

For Examiner's Use	
Question	**Marks**
1	**/ 17**
2	**/ 15**
3	**/ 12**
4	**/ 11**
Total	**/ 55**

This document consists of **18** printed pages and **2** blank pages.

[Turn Over

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

1 Determination of the concentration of hydrogen peroxide and the relative formula mass of hydrated halotrichite

Halotrichite is a mineral which is formed by the weathering and decomposition of pyrite commonly found near volcanic vents. Halotrichite contains Fe^{2+}, one other cation and one anion.

Both potassium manganate(VII) and hydrogen peroxide are oxidising agents which will oxidise the Fe^{2+} in halotrichite to Fe^{3+} under acidic conditions.

equation 1 $MnO_4^-(aq) + 5Fe^{2+}(aq) + 8H^+(aq) \longrightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l)$

equation 2 $2Fe^{2+}(aq) + H_2O_2(aq) + 2H^+(aq) \longrightarrow 2Fe^{3+}(aq) + 2H_2O(l)$

You may assume that the other cation and the anion present in halotrichite do not react with potassium manganate(VII) and hydrogen peroxide.

FA 1 is a solution formed by dissolving 22.26 g of hydrated halotrichite in 100 cm^3 of water.
FA 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 3 is aqueous hydrogen peroxide, H_2O_2.
FA 4 is 0.800 mol dm^{-3} sulfuric acid, H_2SO_4.

Note: FA 1 and FA 4 are also used in questions 3 and 2 respectively.

In *Experiment 1*, you will perform the titration of **FA 1** against potassium manganate(VII), **FA 2**. Analysis of your titration results will enable you to determine the relative formula mass of hydrated halotrichite.

In *Experiment 2*, you will add different volumes of hydrogen peroxide, **FA 3**, to identical samples of **FA 1**. In each case, the amount of hydrogen peroxide you add will only partially oxidise the Fe^{2+} ions present. You will then complete the redox reaction of each mixture by titration with potassium manganate(VII), **FA 2**. Graphical analysis of your results will enable you to determine the concentration of the hydrogen peroxide in **FA 3**.

(a) (i) ***Experiment 1***

1. Fill a burette labelled 'FA 2' with **FA 2**.
2. Use a pipette to transfer 10.0 cm^3 of **FA 1** into a 100 cm^3 conical flask.
3. Use a measuring cylinder to add 5 cm^3 of **FA 4** to the conical flask.
4. Run **FA 2** from the burette into the flask. The end-point is reached when the solution first becomes a permanent pale pink or orange colour.
5. Record your titration results, to an appropriate level of precision, in Table 1.1.
6. Repeat points 2 to 5 until consistent titre values are obtained.

Table 1.1

final burette reading / cm^3					
initial burette reading / cm^3					
volume of **FA 2** used / cm^3					

[2]

(ii) From your titration results, obtain a suitable volume of **FA 2**, V_1, to be used in your calculations. Show clearly how you obtained this volume.

V_1 = …….. cm^3 [2]

(b) **(i)** Use your value for V_1 from **(a)(ii)** to calculate the concentration of iron(II) ions, $[Fe^{2+}]$, in **FA 1**.

$[Fe^{2+}]$ = …….. mol dm^{-3} [2]

(ii) Each formula unit of the hydrated halotrichite contains only one Fe^{2+}.

Calculate the relative formula mass of the hydrated halotrichite.

relative formula mass = …….. [1]

(c) ***Experiment 2***

Each titration is to be performed **once only**, so great care should be taken that you do not overshoot the end-points.

1. Fill a second burette with **FA 3**.
2. Use a pipette to transfer 10.0 cm^3 of **FA 1** into a 100 cm^3 conical flask.
3. Use a measuring cylinder to add 5 cm^3 of **FA 4** to the conical flask.
4. Add, from the second burette, 6.00 cm^3 of **FA 3** to the conical flask.
5. Run **FA 2** from the burette into the flask. The end-point is reached when the solution first becomes a permanent pale pink or orange colour.
6. Record your titration results, to an appropriate level of precision, in Table 1.2.
7. Repeat points 2 to 6 using two other suitable volumes of **FA 3**. Your selected volumes of **FA 3** must not exceed 20.00 cm^3 and should differ by at least 6.00 cm^3.

Table 1.2

volume of **FA 3** added / cm^3	0.00			
final burette reading for **FA 2** / cm^3				
initial burette reading for **FA 2** / cm^3				
volume of **FA 2** used / cm^3				

↑ [2]

enter your value for V_1 from **(a)(ii)** in ***Experiment 1***

(d) Plot a graph of volume of **FA 2** used, on the *y*-axis, against volume of **FA 3** added, on the *x*-axis, on the grid in Fig. 1.1.

Your scale on the *x*-axis should allow you to determine by extrapolation, V_2, which is the volume of **FA 3** required to react completely with 10.0 cm^3 of **FA 1** in the absence of **FA 2**.

Draw a best-fit straight line taking into account all of your plotted points.

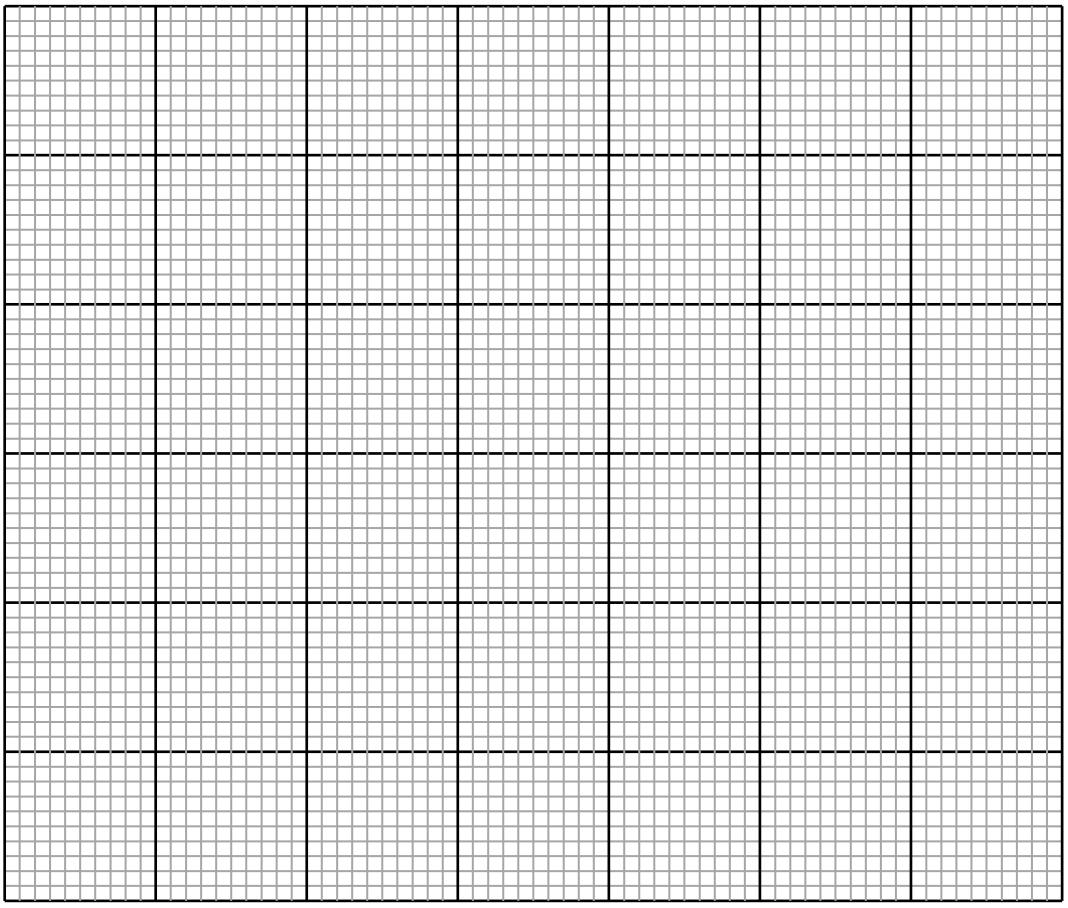


Fig. 1.1

[3]

(e) **(i)** From your graph in **(d)**, determine V_2, which is the volume of **FA 3** required to react completely with the Fe^{2+} present in 10.0 cm^3 of **FA 1** in the absence of **FA 2**.

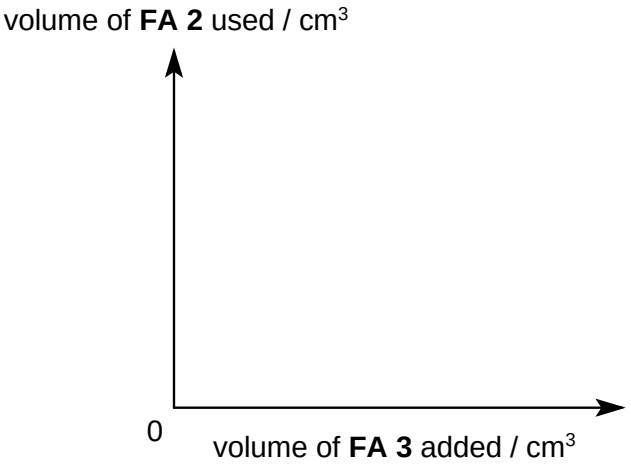
V_2 = …….. cm^3 [1]

(ii) Calculate the concentration of hydrogen peroxide, $[H_2O_2]$, in **FA 3**.

$[H_2O_2]$ = …….. mol dm^{-3} [2]

(f) Sketch the graph which you plotted in **(d)** on the axes given below. Label the graph as **(d).**

On the same axes, sketch the graph that would be obtained if the concentration of $KMnO_4$ in **FA 2** is higher than 0.0200 mol dm^{-3}. Label the graph as **(f)**.



Explain your answer.

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……..……..

……..……..

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……..……..

……..... [2]

[Total: 17]

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2 Determination of the relative atomic mass, A_r, of M in a metal hydroxide, $M(OH)_x$, and the enthalpy change of neutralisation, ΔH_{neut}, of the reaction between H_2SO_4 and $M(OH)_x$

FA 4 is 0.800 mol dm^{-3} sulfuric acid, H_2SO_4.
FA 5 is 1.20 mol dm^{-3} metal hydroxide, $M(OH)_x$.

You will carry out a series of experiments where different volumes of **FA 4** and **FA 5** are mixed together while keeping the total volume of solution constant. You will determine the temperature change, ΔT, for each experiment and plot a graph of ΔT against volume of **FA 5** used.

Using the graph, you will determine the relative atomic mass, A_r, of M in $M(OH)_x$ and the enthalpy change of neutralisation, ΔH_{neut}, for the reaction between sulfuric acid and $M(OH)_x$.

(a) Procedure

1. Place a clean and dry polystyrene cup inside a second polystyrene cup which is placed in a 250 cm^3 glass beaker to prevent the cups from tipping over. Use a 50 cm^3 measuring cylinder to transfer 10.0 cm^3 of **FA 4** into the polystyrene cup and put the lid on.
2. Insert a thermometer through the lid and ensure that the bulb of the thermometer is in contact with **FA 4**. Tilt the cup if necessary to ensure that the bulb of the thermometer is fully immersed. Use the thermometer to measure the temperature of **FA 4**. The temperature, T_1, is the initial temperature of **FA 4**. Record T_1 to 0.1 °C.
3. Measure 40.0 cm^3 of **FA 5** using another 50 cm^3 measuring cylinder.
4. Open the lid of the cup and transfer the 40.0 cm^3 of **FA 5** into the polystyrene cup containing **FA 4**. Replace the lid quickly.
5. Using the thermometer, stir the mixture continuously until it reaches its **highest** temperature. Record this temperature, T_2, to 0.1 °C.
6. Discard the contents of the polystyrene cup. Wash and dry it carefully.
7. Repeat steps 1 to 6 using 15.0 cm^3, 20.0 cm^3, 25.0 cm^3, 30.0 cm^3 and 35.0 cm^3 of **FA 4**, each time using an appropriate volume of **FA 5** so that the total volume of each mixture is 50.0 cm^3.
8. Record all measurements of volume, temperature and temperature change, ΔT, in an appropriate format in the space provided below.

Results

[3]

(b) Plot a graph of temperature change, ΔT, on the *y*-axis, against volume of **FA 5** added, on the *x*-axis, on the grid in Fig. 2.1.

Draw two best-fit straight lines for the points plotted, and extrapolate both lines to find
- the maximum temperature change, ΔT_{max},
- the volume of **FA 5** required to completely neutralise **FA 4**.

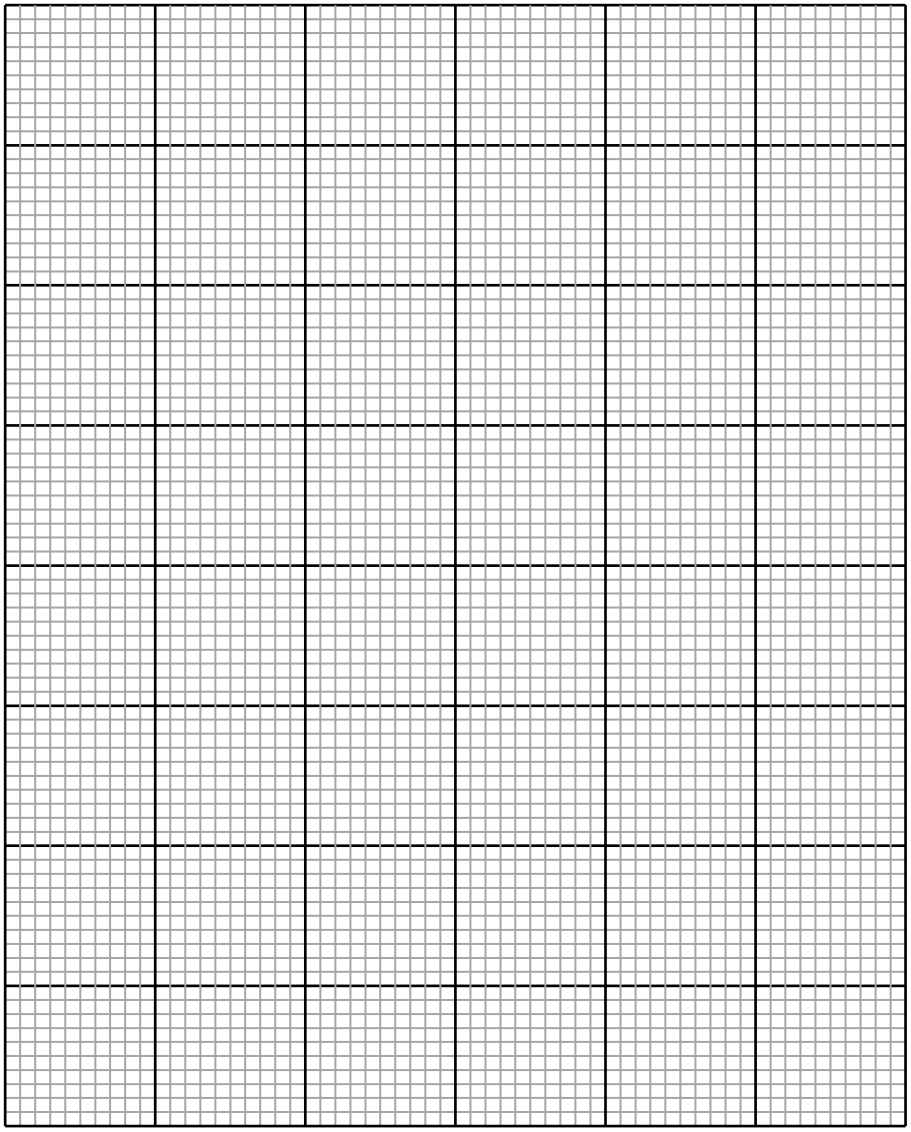

Fig. 2.1

ΔT_{max} = ... ^{o}C

volume of **FA 5** required for complete neutralisation = ... cm^3

Hence, volume of **FA 4** required for complete neutralisation = ... cm^3
[5]

(c) Using your answers to **(b)**, calculate the amount of H_2SO_4 in **FA 4** and $M(OH)_x$ in **FA 5** required for complete neutralisation.

amount of H_2SO_4 in **FA 4** = …….....

amount of $M(OH)_x$ in **FA 5** = …….....
[1]

(d) Hence, suggest the value of x.

value of x = ……..[1]

(e) Given that **FA 5** is prepared by dissolving 16.8 g of solid $M(OH)_x$ in 250 cm^3 of deionised water, calculate the relative atomic mass of M.
[A_r: H, 1.0; O, 16.0]

relative atomic mass of M = ……..... [1]

(f) Calculate the enthalpy change of neutralisation, ΔH_{neut}, for the reaction between H_2SO_4 and $M(OH)_x$.

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

ΔH_{neut} = ……. [2]

(g) State and explain the effect on the magnitude of ΔT_{max} if ethanedioic acid of the same concentration was used instead of sulfuric acid in the experiment in **(a)**.

effect on the magnitude of ΔT_{max} ……

explanation ……

……

……

……..... [2]

[Total: 15]

3 Investigation of some inorganic reactions

FA 1 is a solution of halotrichite. It contains Fe^{2+}, a metal ion **X** and an anion **Y**.

You are to identify **X** and **Y** present in **FA 1**.

Carry out the following tests. Carefully record your observations in Tables 3.1 and 3.2.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Test and identify any gases evolved.

(a) Identification of metal ion X

Table 3.1

	test	observations
(i)	1. Using a measuring cylinder, transfer 10 cm^3 of **FA 1** to a clean boiling tube. 2. Using another measuring cylinder, measure out 10 cm^3 of aqueous sodium carbonate. 3. Use a dropping pipette to transfer 1.0 cm^3 of aqueous sodium carbonate from the measuring cylinder to the boiling tube. 4. Mix the contents of the boiling tube thoroughly. 5. Repeat points 3 and 4 until the measuring cylinder is empty.	
(ii)	Add 1 cm depth of **FA 1** to a test-tube. Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen. Swirl and filter the mixture, collecting the filtrate in a test-tube. The filtrate is **FA 6** which should be retained for use in **(iii)**.	

	test	observations
(iii)	Add 1 cm depth of **FA 6** to a test-tube. Add dilute hydrochloric acid slowly, with shaking, until no further change is seen.	

[5]

(iv) Identify the metal ion **X** in **FA 1**. Use evidence from your observations to support your deduction.

X is ……

evidence ……

……

……

[2]

(b) Identification of anion Y

Table 3.2

	test	observations
(i)	Add 1 cm depth of **FA 1** to a test-tube. Add 5 drops of aqueous barium chloride. Then add dilute hydrochloric acid slowly, with shaking, until no further change is seen.	

[1]

(ii) Hence, identify the anion **Y** in **FA 1**.

Y is …… [1]

(c) Planning – Separation of two cations in solution Z

You are **not** provided with solution **Z**.

Solution **Z** is a solution containing the cations Cu^{2+} and Cr^{3+}.

Plan a procedure to separate the two cations Cu^{2+} and Cr^{3+} in solution **Z** so that each cation is present in separate precipitates.

You may assume that you are provided with:

- aqueous sodium hydroxide, NaOH,
- aqueous ammonia, NH_3,
- dilute hydrochloric acid, HC*l*,
- deionised water,
- the equipment normally found in a school or college laboratory.

In your plan you should:

- **only** be using the chemicals provided, but **not** necessarily all of them
- give brief details of the quantities of chemicals used
- identify by formulae the precipitates obtained after each separation step.

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……... [3]

[Total: 12]

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4 Planning

Calcium hydroxide, $Ca(OH)_2$, is a sparingly soluble salt. In a saturated solution containing some undissolved solid $Ca(OH)_2$, the following equilibrium is established.

$$Ca(OH)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2OH^-(aq)$$

The approximate solubilities of $Ca(OH)_2$ in deionised water and in 0.020 mol dm^{-3} aqueous sodium hydroxide at 25 ºC are given in Table 4.1.

Table 4.1

	approximate solubility of $Ca(OH)_2$ at 25 ºC / mol dm^{-3}
deionised water	0.025
0.020 mol dm^{-3} aqueous sodium hydroxide, NaOH	0.005

(a) Explain why the solubility of $Ca(OH)_2$ is lower in 0.020 mol dm^{-3} aqueous NaOH than in water.

……..…….....

……..... [1]

(b) In this question, you are to plan a procedure that allows you to determine the solubility of $Ca(OH)_2$ in 0.020 mol dm^{-3} aqueous sodium hydroxide and the solubility product of $Ca(OH)_2$ at 25 °C.

The solubility and solubility product, K_{sp}, of $Ca(OH)_2$ can be determined by titrating a saturated solution of $Ca(OH)_2$ against dilute hydrochloric acid placed in a burette.

You should plan to prepare:

- 150 cm^3 of **FA 7**, a saturated solution of $Ca(OH)_2$ in 0.020 mol dm^{-3} aqueous sodium hydroxide at 25 °C, separated from any undissolved solid,
- 250 cm^3 of **FA 8**, a solution of hydrochloric acid of a suitable concentration for titration.

You may assume that you are provided with:

- solid calcium hydroxide, $Ca(OH)_2$,
- 0.020 mol dm^{-3} aqueous sodium hydroxide, NaOH,
- 0.250 mol dm^{-3} dilute hydrochloric acid, HC*l*,
- methyl orange,
- the equipment normally found in a school or college laboratory.

In your plan you should include brief details of:

- the apparatus you would use,
- the procedure you would follow,
- the measurements you would make,
- how you would determine the solubility of $Ca(OH)_2$ in 0.020 mol dm^{-3} aqueous sodium hydroxide and the solubility product of $Ca(OH)_2$ at 25 °C.

[10]

[Total: 11]

Qualitative Analysis notes
[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	***reaction with***	
	NaOH(aq)	NH_3(aq)
aluminium, Al^{3+}(aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH_4^+(aq)	ammonia produced on heating	–
barium, Ba^{2+}(aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca^{2+}(aq)	white ppt. with high [Ca^{2+}(aq)]	no ppt.
chromium(III), Cr^{3+}(aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu^{2+}(aq)	pale blue ppt. insoluble in excess	blue-ppt. soluble in excess giving dark blue solution
iron(II), Fe^{2+}(aq)	green ppt., turning brown on contact with air insoluble in excess	green ppt., turning brown on contact with air insoluble in excess
iron(III), Fe^{3+}(aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
magnesium, Mg^{2+}(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn^{2+}(aq)	off-white ppt., rapidly turning brown on contact with air insoluble in excess	off-white ppt., rapidly turning brown on contact with air insoluble in excess
zinc, Zn^{2+}(aq)	white ppt. soluble in excess	white ppt. soluble in excess

(b) Reactions of anions

anion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives pale cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq))
nitrate, NO_3^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (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