

CHEMISTRY

Higher 2

Paper 4 Practical

2 September 2020 2 hours 30 minutes

9729/04

Candidates answer on the Question paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name, class and exam index number on all the work you hand in.

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

Write in dark blue or black pen.

You may use a HB pencil for any diagrams, graphs.

Do not use staples, paper clips, glue or correction fluid.

Answer **all** questions in the spaces provided on the Question Paper. **Do not spend more than 40 minutes on Q1 and Q2.**

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 18 and 19.

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.

Shint	
Laboratory	

Chift

For Examiner's Use	
1	/8
2	/9
3	/22
4	/16
Total	/55

This document consists of 19 printed pages.

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

1 Inorganic Qualitative Analysis

FA 1 is a solid sample which contains two cations and one anion. One of the cations is Fe^{3+} .

Perform the tests described in Table 1.1 and record your observations in the table. Test and identify any gases evolved.

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

Table 1.1

		tests	observations
(a)	(i)	Add one spatula full of FA 1 into a test-tube. Place about 5 cm depth of deionised water into the test-tube and shake the mixture until all the solid has dissolved.	
		Use this FA 1 solution for tests (ii) and (iii).	
	(ii)	To a 2 cm depth of FA 1 solution in a test-tube, add one spatula of solid sodium carbonate.	
	(iii)	To a 1 cm depth of FA 1 solution in a boiling tube, add aqueous sodium hydroxide dropwise, with shaking, until it is in excess. Gently warm the mixture.	
		Then filter the mixture, collecting the filtrate in a clean test-tube. Keep the filtrate for test (iv) .	
	(iv)	To a 1 cm depth of the filtrate from test (iii) in a test tube, add BaC <i>l</i> ₂ (aq) dropwise. Then add dilute hydrochloric acid until no further change.	

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(b)	(i)	By considering the observations made in Table 1.1 and making reference to the <i>Qualitative Analysis Notes</i> , identify the other cation and the anion present in FA 1 .		For Examiner's Use
		The other cation in FA1 is		
		The anion in FA 1 is	[2]	
	(ii)	By considering the ions present in FA 1 , suggests an explanation for observations made in test (a)(ii) .		
			[1]	

[Total: 8]

2 Organic Qualitative Analysis

In this question, you will deduce the structure of an organic compound, FA 2. The molecular formula of FA 2 is $C_4H_8O_2$.

Perform the tests described in Table 2.1, and record your observations in the table. Test and identify any gases evolved. Some observations have been recorded.

Do not use Bunsen burner for heating. Use the hot water provided.

	tests	observations
(i)	Place about 2 cm depth of aqueous sulfuric acid in a test-tube.	
	To this test-tube, add 10 drops of FA 2 , followed by 1 drop of aqueous potassium manganate(VII).	
	Warm the mixture in the hot water bath for two minutes.	
(ii)	Place about 2 cm depth of aqueous silver nitrate in a test-tube.	
	To this test-tube, add 10 drops of aqueous sodium hydroxide, followed by aqueous ammonia until a clear solution is formed.	
	To this test-tube, add 10 drops of FA 2 .	
	Warm the mixture in the water bath for two minutes.	
(iii)	Place about 1 cm depth of deionised water in a test-tube. To this test-tube, add 10 drops of FA 2 and add 10 drops of aqueous sodium hydroxide. Now add iodine solution dropwise, until a permanent yellow/ orange colour is obtained.	
	Warm the mixture in the water bath for two minutes.	
(iv)	Place about 1 cm depth of FA 2 in a test-tube.	Effervescence observed.
	To this test-tube, cautiously add a small piece of sodium metal.	H ₂ gas produced extinguishes a lighted splint with a "pop" sound.
(v)	Place about 1 cm depth of FA 2 in a test-tube.	
	To this test-tube, add 2,4-dinitrophenylhydrazine dropwise.	Orange ppt. formed

Table 2.1

[3]

5

(b)	(i)	Using the observations in (a)(ii) and (v) in Table 2.1, identify one functional group present in FA 2 . Give evidence(s) to support your conclusion.		For Examiner's Use
		Functional group:		
		Evidence:		
			[2]	
	(ii)	Using the observations in other tests in Table 2.1, identify the other functional group present in FA 2 .		
		Give evidence(s) to support your conclusion.		
		Functional group:		
		Evidence:		
			[2]	
	(iii)	Suggest two possible structures of FA 2 .		

[2]

[Total: 9]

3 Investigation of reaction between manganate(VII) ions and ethanedioate ions.

FA 3 is 0.0100 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃

- **FA 4** is 0.0200 mol dm⁻³ potassium manganate(VII), KMnO₄
- $\textbf{FA 5} \quad \text{is } 0.200 \text{ mol } dm^{-3} \text{ ethanedioic acid}, \ H_2 C_2 O_4$
- **FA 6** is 1.0 mol dm⁻³ sulfuric acid, H₂SO₄
- **FA7** 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 4**, **FA 5** and **FA 6**. 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.

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 8**, 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 3 added)
- decimal time, **t**, which is the transfer time converted to minutes in 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 **FA7** to each of the labelled boiling tubes, **1** to **5**.
- 2. Fill a burette with FA 3.

Preparing the reaction mixture

- 3. Use appropriate measuring cylinders to add to the beaker labelled reaction mixture
 - 50.0 cm³ of **FA 5**,
 - 5.0 cm³ of **FA 6**,
 - 45.0 cm³ of deionised water.
- 4. Place 25.0 cm³ of **FA 4** into a 25 cm³ measuring cylinder.
- 5. At a convenient time, pour **FA 4** 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

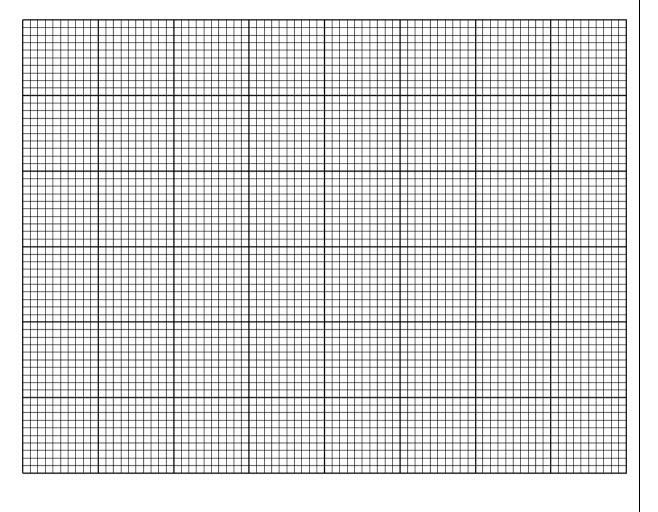
- **Note:** You will only be able to perform each titration **once**. Be careful not to overshoot the end-point.
- 9. Pour all the contents of boiling tube **1** into a clean conical flask. Rinse this boiling tube with deionised water and add the washings to the conical flask.
- 10. Record the initial burette reading. Titrate the I₂ in this solution with **FA 3** 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 3** added.
- 11. Wash this conical flask thoroughly with water.
- 12. Top up the burette with FA 3.
- 13. Repeat points 9 to 11 as required for each of the remaining boiling tubes.

(b) Experimental results

[5]

(c) (i) On the grid below, plot a graph of the volume of sodium thiosulfate, **FA 3**, on the *y*-axis, against time, **t**, on the *x*-axis.

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



[3]

For

Examiner's

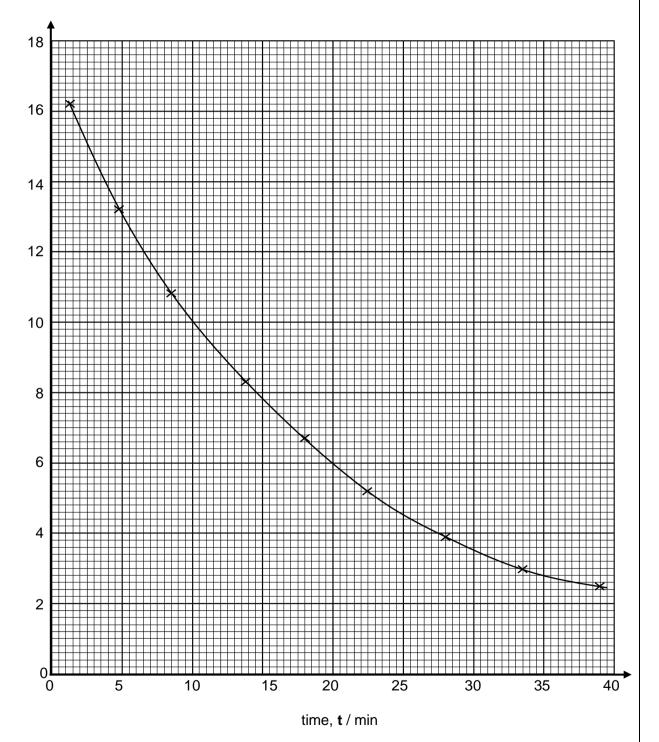
Use

(ii) Rate of reaction at time **t** is indicated by the slope of 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.

[2]

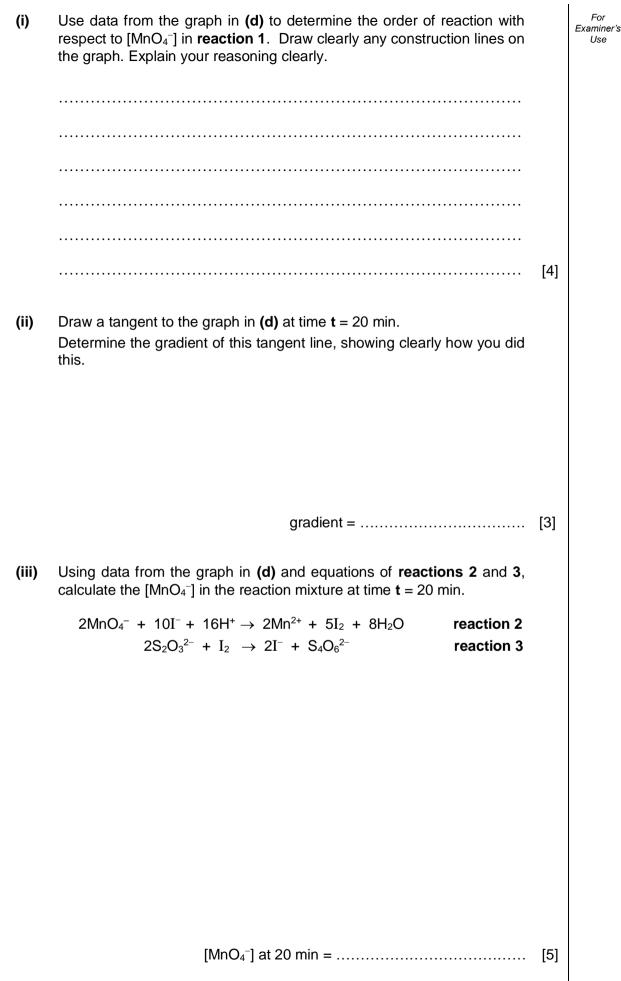
A student performed a similar experiment in cooler conditions. In point 3, she used the same volumes of **FA 5** and **FA 6** that you used but she also added 5.0 cm³ of a solution of manganese(II) sulfate, MnSO₄, a source of Mn²⁺ to catalyse the reaction. She only added 40.0 cm³ of deionised water, 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.



Volume of **FA3** / cm³

10



[Total: 22]

(d)

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[Turn Over

4 Planning

When sodium hydrogencarbonate, NaHCO₃, is heated, it decomposes to form sodium carbonate, Na_2CO_3 .

$$2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(l) + CO_2(g) \Delta H_r$$

The enthalpy change of reaction, ΔH_r , for decomposition of NaHCO₃ cannot be determined directly and so an indirect method is used.

By determining the enthalpy change of reaction between Na₂CO₃(s) and hydrochloric acid, ΔH_1 , and that between NaHCO₃(s) and hydrochloric acid, ΔH_2 , you can obtain a value for ΔH_r for decomposition of NaHCO₃.

Experiment 1 Reaction between sodium carbonate, Na₂CO₃(s), and dilute hydrochloric acid, HC*l*(aq)

$$Na_2CO_3(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g)$$
 ΔH_2

You are provided with

FA 8 2.0 mol dm⁻³ hydrochloric acid, HC*l*(aq)

FA9 a pre-weighed sample of solid sodium carbonate, Na₂CO₃(s)

You are to carry out an experiment following the method below and use the results of the experiment to determine the enthalpy change of reaction between Na₂CO₃(s) and HC*l*(aq), ΔH_1 .

Read through the method. In an appropriate format in the space provided on the next page, prepare suitable tables to record your results.

Method

- 1. Weigh the stoppered weighing bottle labelled **FA 9** and its contents. Record the total mass of weighing bottle and **FA 9**.
- 2. Support the Styrofoam cup in the 250 cm³ beaker.
- 3. Use a 50 cm³ measuring cylinder to transfer 50.0 cm³ of **FA 8** into the Styrofoam cup. **FA 8** is in excess.
- 4. Place the thermometer in this **FA 8** and record the initial temperature.
- 5. **Carefully** add all the **FA 9**, in small portions to avoid spray, from the weighing bottle to **FA 8** in the Styrofoam cup. While adding the solid, stir the mixture constantly with the thermometer. Record the maximum temperature reached.
- 6. Calculate and record the maximum temperature change, ΔT , that occurred during the reaction.
- 7. Reweigh the stoppered weighing bottle with any residual **FA 9** and record the mass.
- 8. Calculate and record the mass of **FA 9** added to the cup.
- 9. Record your results on the next page.

For

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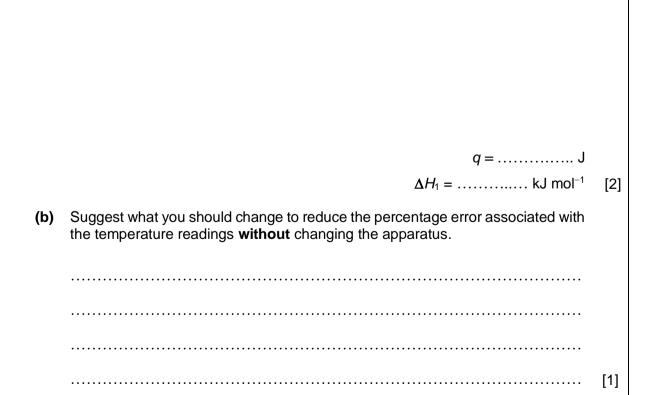
[3]

(ii) Use your results in (a)(i) to calculate the heat change, q, for your experiment and hence determine a value for ΔH_1 .

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 $Na_2CO_3(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g) \Delta H_1$

Assume that the specific heat capacity of the final solution is 4.20 J g⁻¹ K⁻¹ and that its density is 1.03 g cm⁻³. [A_r : Na, 23.0; C, 12.0; O, 16.0]



For Examiner's Use

Experiment 2 Reaction between sodium hydrogencarbonate, NaHCO₃(s), and dilute hydrochloric acid, HC*l*(aq)

 $NaHCO_3(s) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l) + CO_2(g) \qquad \Delta H_2 > 0$

The reaction between $NaHCO_3(s)$ and HCl(aq) is an endothermic process.

The maximum temperature change occurring during this reaction may be determined graphically.

The maximum temperature change, ΔT_{max} , obtained from the graph can be used to calculate the heat change, q, for this experiment. Using q, a value for ΔH_2 may be determined.

You may assume that you are provided with

- 150 cm³ of 2 mol dm⁻³ hydrochloric acid, HCl(aq)
- 10 g of solid sodium hydrogencarbonate, NaHCO₃(s)
- the equipment normally found in a school or college laboratory.
- (c) (i) Outline how you would carry out an experiment to determine the maximum temperature change, ΔT_{max} , graphically for the reaction between NaHCO₃(s) and HC*l*(aq) in order to determine an accurate and reliable value for ΔH_2 .

Your answer should include brief details of:

- the apparatus you would use,
- the quantities you would use, so that there is an excess of HCl(aq),
- the procedure you would follow,
- the measurements you would make to allow a suitable temperaturetime graph to be drawn,
- how you would ensure that an accurate and reliable value of ΔT_{max} is obtained.

 [5]

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(ii) Sketch, on Fig. 4.1, the graph you would expect to obtain using the measurements you planned to make in (c)(i).

Indicate how the maximum temperature change, ΔT_{max} , can be determined on the graph.

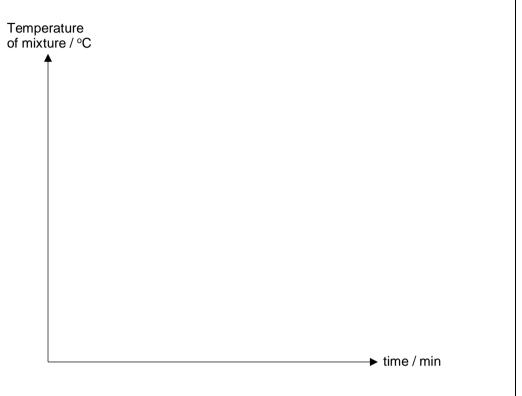


Fig. 4.1

(d) Assume that:

$Na_2CO_3(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g)$	$\Delta H_1 = -26.7 \text{ kJ mol}^{-1}$
$NaHCO_3(s) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l) + CO_2(g)$	$\Delta H_2 = +24.2 \text{ kJ mol}^{-1}$

Use these values of ΔH_1 and ΔH_2 to determine a value for enthalpy change of reaction, ΔH_r , for decomposition of NaHCO₃(s).

 $2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(l) + CO_2(g) \Delta H_r$

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[2]

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(e) The maximum temperature change, ΔT_{max} , can also be determined by direct measurement of the initial temperature and the final temperature reached only. Explain why the graphical method is likely to give a more accurate value of ΔT_{max} than the direct measurement method.

[Total: 16]

Qualitative Analysis Notes

(a) Reactions of Aqueous Cations

	Reaction with		
cation	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, 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), Cu²+(aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution	
iron(II), Fe²+(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, Zn²+(aq)	white ppt. soluble in excess	white ppt. soluble in excess	

Anion	Reaction
carbonate, CO_3^{2-}	CO ₂ liberated by dilute acids
choride, C <i>l</i> ⁻ (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, NO₃⁻(aq)	NH_3 liberated on heating with $OH^-(aq)$ and Al foil
nitrite, NO₂⁻(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 on warming with dilute acids; gives white ppt. with Ba ²⁺ (aq) (soluble in excess dilute strong acids)

(c) Tests for Gases

gas	Test and test results
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 acidified aqueous 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