



CANDIDATE
NAME

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CIVICS
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CHEMISTRY

Paper 4 Practical

9729/04

31 August 2021

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, civics group and registration number on 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 an HB pencil for any diagrams or graphs.
Do not use paper clips, highlighters, 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 23 and 24.

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	/ 22
2	/ 22
3	/ 11
Total	/ 55

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

For
Examiner's
Use

1 Investigation on the behaviour of acids and bases in aqueous solution

FA 1 is 1.00 mol dm⁻³ sodium hydrogencarbonate, NaHCO₃

FA 2 is sodium hydroxide, NaOH, of concentration between 1.5 mol dm⁻³ and 2.0 mol dm⁻³

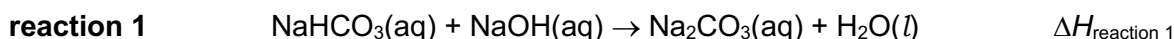
FA 3 is 2.00 mol dm⁻³ sulfuric acid, H₂SO₄

According to the Arrhenius theory, an acid-base neutralisation reaction involves reacting together H⁺(aq) and OH⁻(aq) to produce water molecules.



This task involves two different acid-base reactions.

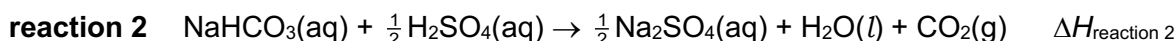
The first reaction is between sodium hydrogencarbonate, **FA 1**, and sodium hydroxide, **FA 2**.



The molar enthalpy change for **reaction 1**, $\Delta H_{\text{reaction 1}}$, is the enthalpy change when 1.00 mol of NaHCO₃ reacts completely with NaOH.

Instead of using an indicator to determine the endpoint, you will perform a thermometric titration to determine the equivalence point of the reaction. The equivalence point is that point where H⁺(aq) from the acid and OH⁻(aq) from the base are present in equal molar amounts.

The second reaction is between sodium hydrogencarbonate, **FA 1**, and sulfuric acid, **FA 3**.



The molar enthalpy change for **reaction 2**, $\Delta H_{\text{reaction 2}}$, is the enthalpy change when 1.00 mol of NaHCO₃ reacts completely with H₂SO₄.

In your first experiment, you will perform a thermometric titration by adding portions of **FA 2** progressively to a known volume of **FA 1**. You will continue adding **FA 2** until the equivalence point is reacted and passed. Throughout the experiment you will note and record the temperature of the mixture after each addition.

You will then analyse your results graphically in order to determine the

- titration volume at the equivalence point, $V_{\text{equivalence}}$,
- maximum temperature change, $\Delta T_{\text{maximum 1}}$,
- molar enthalpy change, $\Delta H_{\text{reaction 1}}$, for **reaction 1**.

In your second experiment, you will mix together a given volume of **FA 1** with a suitable volume of **FA 3**.

You will then determine the

- maximum temperature change, $\Delta T_{\text{maximum 2}}$,
- molar enthalpy change, $\Delta H_{\text{reaction 2}}$, for **reaction 2**.

(a) The reaction between FA 1 and FA 2

In this task you will need to record the maximum temperature of the reaction mixture when specified volumes of **FA 2** have been added. It is important that the volume of **FA 2** recorded is the total volume you have added up to that point when the temperature reading was made.

Note: If you overshoot on an addition, record the **actual** total volume of **FA 2** added up to that point.

In an appropriate format in the space provided on page 4, record all values of temperature, T , and each total volume of **FA 2** added.

- (i)
1. Fill a burette with **FA 2**.
 2. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
 3. Using a pipette, transfer 25.0 cm^3 of **FA 1** to the first Styrofoam cup.
 4. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature.
 5. From the burette, add 2.00 cm^3 of **FA 2** to the cup and stir the mixture thoroughly.
 6. Read and record the maximum temperature of the mixture, T , and the volume of **FA 2** added.
 7. Repeat points 5 and 6 until a total of 30.00 cm^3 of **FA 2** has been added. After each addition of **FA 2**, record the maximum temperature of the mixture and the total volume of **FA 2** added up to that point.

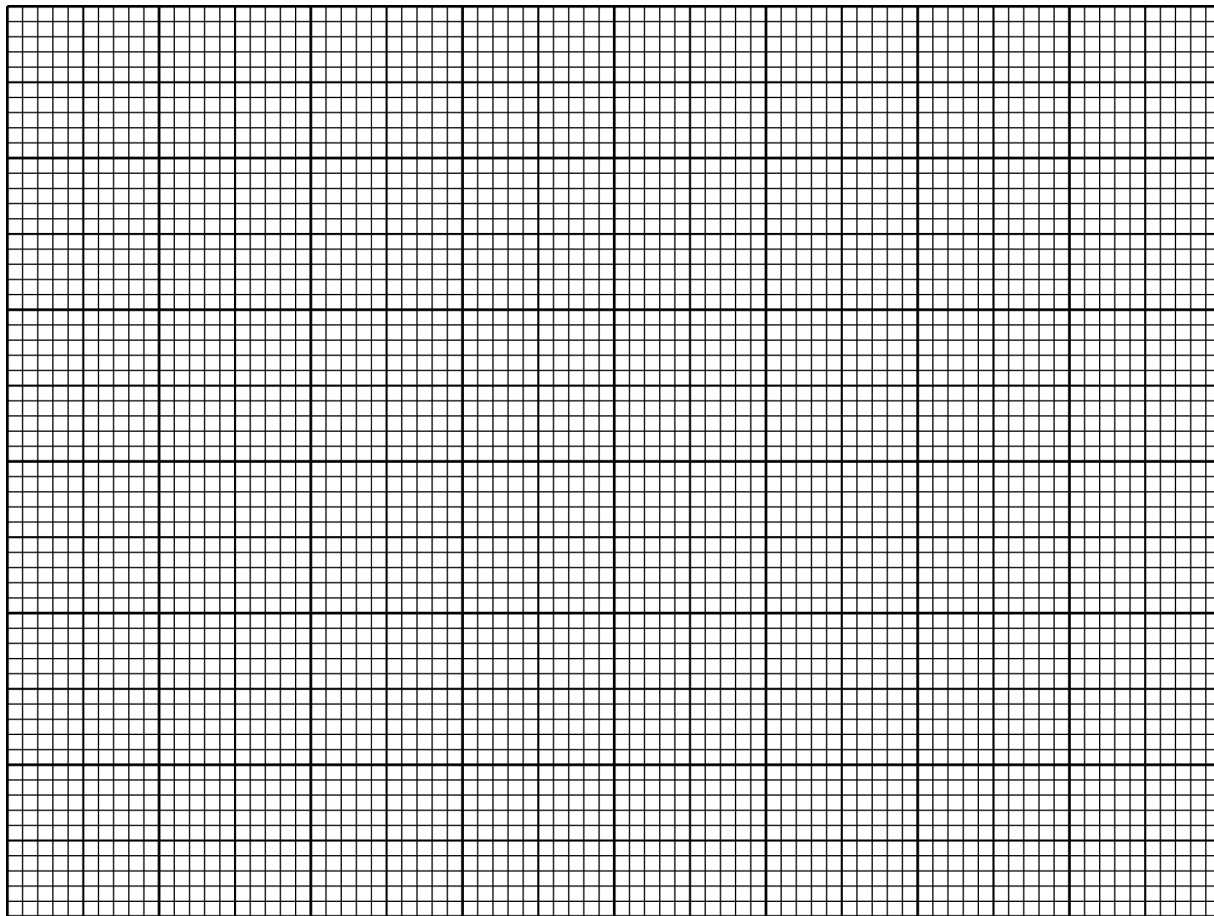
Results

*For
Examiner's
Use*

[2]

- (ii) On the grid below, plot a graph of temperature, T (y -axis) against volume of **FA 2** added (x -axis).

For
Examiner's
Use



[4]

- (iii) Draw **two** smooth lines of best fit.

- The first best-fit line should be drawn using the plotted points where the temperature is **rising**.
- The second best-fit line should be drawn using the plotted points where the temperature is **falling**.
- Extrapolate these lines until they cross.

Note: Each line should have a shape best suited to its plotted points.

(iv) Determine from your graph

- the maximum temperature reached, T_{maximum} ,
- the maximum temperature change, $\Delta T_{\text{maximum 1}}$,
- the volume, $V_{\text{equivalence}}$, of **FA 2** needed to completely react with 25.0 cm³ of **FA 1**.

Show on your graph how you obtained these values.

Record these values in the spaces provided below.

maximum temperature reached, $T_{\text{maximum}} =$

maximum temperature change, $\Delta T_{\text{maximum 1}} =$

volume of **FA 2** used, $V_{\text{equivalence}} =$

[3]

(b) Ideally, the graph line **before** the equivalence point should be an *increasing* curve that *concaves downward* (that is, gradient of the curve decreases).

(i) Explain why the graph line is increasing.

.....

[1]

(ii) Explain why the graph line concaves downward.

.....

[1]

(c) The reaction between FA 1 and FA 3

The molar enthalpy change for **reaction 2**, $\Delta H_{\text{reaction 2}}$, is the enthalpy change when 1.00 mol of NaHCO_3 reacts completely with H_2SO_4 .

In this task you will calculate a value for the molar enthalpy change, $\Delta H_{\text{reaction 2}}$. To do this, you will need to determine the maximum temperature change produced when measured volumes of **FA 1** and **FA 3** are mixed.

Upon considering the concentrations of **FA 1** and **FA 3**, 15 cm³ of **FA 3** was chosen to add to 40 cm³ of **FA 1**.

Note: You should be aware that the reaction mixture will produce considerable frothing.

- (i) Explain why 15 cm³ of **FA 3** was chosen to add to 40 cm³ of **FA 1**.

.....

[1]

- (ii) Identify the apparatus you intend to use to measure the volume of **FA 1**.

Explain your choice.

apparatus

explanation

.....

[1]

(iii) In an appropriate format in the space provided below, record all values of measured temperature for this experiment.

For
Examiner's
Use

1. Place a Styrofoam cup inside a second Styrofoam cup which is held in a glass beaker to prevent it tipping over.
2. Transfer 40 cm³ of **FA 1** into the first Styrofoam cup using the apparatus identified in **1(c)(ii)**.
3. Stir the **FA 1** solution in the cup with the thermometer. Read and record its temperature, $T_{\text{FA 1}}$. This is the initial temperature of **FA 1**.
4. Wash and dry the thermometer.
5. Measure 15.0 cm³ of **FA 3** using an appropriate measuring cylinder. Stir the **FA 3** solution in the measuring cylinder with the thermometer. Read and record its temperature, $T_{\text{FA 3}}$. This is the initial temperature of **FA 3**.
6. Carefully add the contents of the measuring cylinder to the Styrofoam cup in **small** portions to avoid too much frothing.
7. Place the lid on the cup and insert the thermometer through the lid. Stir the mixture.
8. Continue to stir the mixture. Measure and record the temperature, T_{mixture} that shows the maximum change from the initial temperature.

Results

[1]

Calculations*For
Examiner's
Use*

- (d) For the purpose of these calculations, you should assume that the reaction mixture has a density of 1.00 g cm^{-3} and a specific heat capacity, c , of $4.18 \text{ J g}^{-1} \text{ K}^{-1}$.

Use your results from **(a)(iv)** to calculate

- (i) the concentration of sodium hydroxide, $[\text{NaOH}]$, in **FA 2**,

$[\text{NaOH}]$ in **FA 2** =[1]

- (ii) the heat change, q , for the reaction occurring in **(a)(i)**, and hence determine a value for the molar enthalpy change for **reaction 1**, $\Delta H_{\text{reaction 1}}$.

q =

$\Delta H_{\text{reaction 1}}$ =[2]

- (e) Use your results from (c)(iii) to calculate a value for the molar enthalpy change for **reaction 2**, $\Delta H_{\text{reaction 2}}$. For the experiment in (c)(iii), the weighted average initial temperature, T_{average} , of **FA 1** and **FA 3** may be calculated using the formula given below.

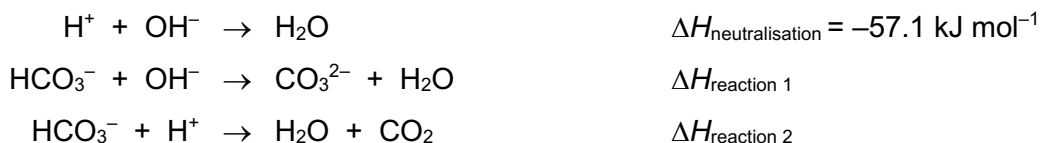
For
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Use

$$T_{\text{average}} = \frac{(V_{\text{FA1}} \times T_{\text{FA1}}) + (V_{\text{FA3}} \times T_{\text{FA3}})}{(V_{\text{FA1}} + V_{\text{FA3}})}$$

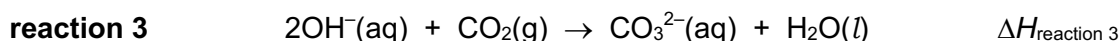
$$q = \dots\dots\dots$$

$$\Delta H_{\text{reaction 2}} = \dots\dots\dots [1]$$

- (f) Ionic equations for neutralisation, **reaction 1**, and **reaction 2** are shown below.



Carbon dioxide may be removed from stale air by bubbling the air through aqueous alkali. An equation for this reaction is given below.



Use your answers to (d)(ii) and (e), together with the molar enthalpy change of neutralisation, $\Delta H_{\text{neutralisation}}$, to determine a value for the molar enthalpy change for this reaction, $\Delta H_{\text{reaction 3}}$.

$$\Delta H_{\text{reaction 3}} = \dots\dots\dots [1]$$

- (g) Another student planned a different thermometric titration experiment to determine the value of the titration volume at the equivalence point, $V_{\text{equivalence}}$, and the maximum temperature change, $\Delta T_{\text{maximum 1}}$, for **reaction 1**.

For
Examiner's
Use

In this other experiment, different volumes of **FA 1** and **FA 2** as shown in Table 1.1 are mixed, and the maximum temperature change, ΔT_{max} , is determined for each mixture.

Table 1.1

experiment	1	2	3	4	5	6	7	8
$V_{\text{FA 2}} / \text{cm}^3$	10.00	15.00	20.00	25.00	30.00	35.00	40.00	45.00
$V_{\text{FA 1}} / \text{cm}^3$	45.00	40.00	35.00	30.00	25.00	20.00	15.00	10.00

ΔT_{max} is plotted against the volume of **FA 2** used, $V_{\text{FA 2}}$. $V_{\text{equivalence}}$ and $\Delta T_{\text{maximum 1}}$ are determined from the intersection of the two straight lines drawn as shown in Fig. 1.1.

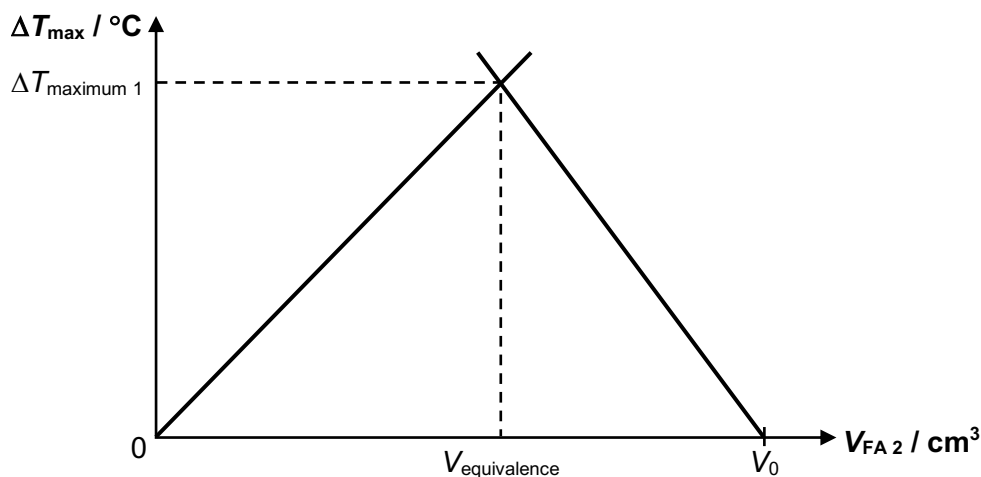


Fig. 1.1

- (i) Suggest a value for V_0 in Fig. 1.1.

$V_0 = \dots\dots\dots \text{cm}^3$ [1]

- (ii) The student claimed that this method gives a more accurate value of $V_{\text{equivalence}}$ and $\Delta T_{\text{maximum 1}}$ compared to the method in 1(a).

Do you agree with the student? Explain.

$V_{\text{equivalence}}$

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$\Delta T_{\text{maximum } 1}$

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[2]

[Total: 22]

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Use*

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Use*

2 Determination of identity of the impurity in FA 4, the decomposition route of NaHCO_3 , the percentage purity of FA 4

Solid FA 4 is anhydrous sodium hydrogencarbonate, NaHCO_3 , of greater than 95% purity that has been contaminated with a sodium halide.

You are also provided with an aqueous solution of **FA 4**, labelled **FA 4 solution**.

(a) You will devise and perform a simple test, based on the Qualitative Analysis Notes on pages 23–24, to identify the halide present in the **FA 4 solution**.

(i) Describe a test, using only the bench reagents provided, which will allow you to identify the halide impurity present in **FA 4**.

.....
.....[1]

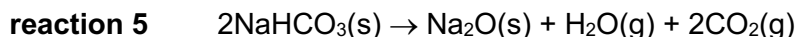
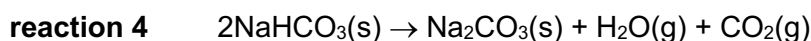
(ii) Perform the test you described in 2(a)(i) using the **FA 4 solution** provided.

Record your observations and hence deduce the identity of the halide in **solid FA 4**.

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.....
identity of halide [2]

(b) Thermal decomposition of NaHCO_3

When NaHCO_3 is heated above 110 °C (but not heated to “red heat”) it has been observed that both $\text{H}_2\text{O}(\text{g})$ and $\text{CO}_2(\text{g})$ are evolved, and that after this decomposition is complete, a white solid residue remains. Among possible reactions, the two following reactions seem the ones that are most likely to explain these facts:



Sodium halides are stable to heat.

In this experiment, solid **FA 4** is heated gently in a boiling tube, over a luminous Bunsen flame, until **all** NaHCO_3 has been decomposed. The data will be used to determine

- the percentage mass loss,
- whether the decomposition takes place *via* **reaction 4** or **reaction 5**,
- the amount of CO_2 lost,
- the percentage purity of NaHCO_3 in **FA 4**

[Ar: Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

1. Weigh accurately between 2.00 g and 2.50 g of **FA 4** in the boiling tube provided. Record your weighings in the space provided below.
2. Heat the tube and content **gently** for 5 minutes.

Use a **luminous** Bunsen flame with the air-hole **closed** for this purpose. Ensure even heating of the sample and boiling tube.

3. Place the boiling tube into a **dry** 250 cm³ beaker to cool.

You may wish to proceed with other experiments while waiting for the boiling tube to cool.

4. Weigh and record the mass of the cooled boiling tube containing the residue.
 5. Repeat points 2 (heat gently for 2 minutes subsequently) to 4 as necessary until a constant mass is obtained.
 6. **Turn off the Bunsen burner.**
- (i) In an appropriate format in the space below, record all weighings.

[4]

- (ii) Using your results, calculate the mass lost upon decomposition of **FA 4**.

mass lost = [1]

- (iii) Using your answer to **2(b)(ii)**, calculate the percentage mass lost.

percentage mass lost = [1]

For
Examiner's
Use

- (iv) Using your answer to **2(b)(iii)**, determine whether the decomposition occurs *via* **reaction 4** or **reaction 5**

For
Examiner's
Use

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.....[2]

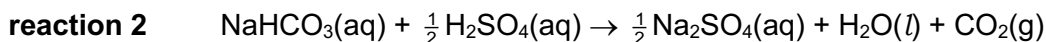
- (v) Using your answer to **2(b)(ii)** and **2(b)(iv)**, calculate the amount of CO_2 lost.

amount of CO_2 lost = [1]

- (vi) Using your answer to **2(b)(v)**, calculate the percentage purity of NaHCO_3 in **FA 4**.

percentage purity of NaHCO_3 in **FA 4** = [2]

The percentage purity of NaHCO₃ in **FA 4** was determined *via* volatilisation gravimetric analysis in **2(b)**. Titrimetric analysis such as the thermometric titration in **1(a)** using **reaction 1** can also be used. In addition, the more usual acid-base titrations based on either **reaction 1** or **reaction 2** are applicable too.



You may assume that you are provided with:

- In your plan, you should include brief details of

- the apparatus you would use,
- the quantities you would use,
- the procedure you would follow,
- the indicator you would use, including the colour change at the endpoint,
- the measurements you would make,
- an outline of how you would use your results to determine the percentage purity of NaHCO_3 in **FA 4**.

[A_r: Na, 23.0; O, 16.0; C, 12.0; H, 1.0]

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This image shows a full page of white paper designed for handwriting practice. It features approximately 20 evenly spaced horizontal dotted lines running from left to right across the entire width of the page. There are no margins, text, or other markings present.

[8]

[Total: 22]

3 Qualitative Analysis

FA 5 contains two unknown cations.

FA 6 contains one unknown anion.

- (a) Perform the tests described in Table 3.1, and record your observations in the table. Test and identify any gases evolved. If there is no observable change, write **no observable change**.

Table 3.1

tests		observations
1.	To 2 cm depth of FA 5 in a test tube, add aqueous sodium hydroxide. Warm the mixture. Filter the mixture and use the resulting filtrate for Test 2 and residue for Test 3 .	
2.	To 1 cm depth of the filtrate from Test 1 , add dilute nitric acid dropwise with shaking until in excess. Add aqueous ammonia.	
3.	Pour nitric acid onto the residue on the filter paper from Test 1 . Divide the liquid that filters through into 2 equal portions. To one portion, add aqueous sodium hydroxide slowly with shaking.	
4.	To the other portion of the liquid from Test 3 , add aqueous ammonia.	
5.	Place about 2 cm depth of the FA 6 solution in a test-tube. To this solution, add about 2 cm depth of FA 5 .	

[6]

(b) Deduce the unknown ions present in the following solutions.

For
Examiner's
Use

(i) FA 5

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.....

.....[2]

(ii) FA 6

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.....[1]

(c) Write equations to account for the observations in **Test 5**.

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.....[2]

[Total: 11]

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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

<i>anion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives pale cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in dilute strong acids)

(c) Tests for gases

<i>gas</i>	<i>test and test result</i>
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

<i>halogen</i>	<i>colour of element</i>	<i>colour in aqueous solution</i>	<i>colour in hexane</i>
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