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\diamond	Name:
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1. Qualitative Analysis

Qualitative Analysis is a method used to identify ions in compounds based on chemical reactions that involve:

- Evolution of gas
- Formation of precipitate
- Colour change

1.1 Safety

In addition to the general lab safety rules, the following should be observed at all times when implementing qualitative analysis experiments:

- Students **must** wear safety goggles.
- **Do not** touch chemicals with your hands. Wash off immediately any chemicals that comes into contact with the skin.
- Note the smell of a gas by wafting it gently to the nose. **Do not** take in deep breath of gas.
- Dispose wastes carefully. **Dilute** concentrated acids and alkalis before pouring them away and **flush** the sink with plenty of water. **Dispose** solid waste into **waste bins**, not the sink.
- A test-tube holder must be used when tests involve:
 - o warming
 - adding concentrated acids, alkalis and flammable reagents. Such experiments should be carried out in the fume cupboard.
- When warming the mixture or burning, direct the mouth of test-tubes away from people.
- Turn off the Bunsen flame when not in use.

2. Manipulative Skills

2.1 Quantity of Chemicals

Collect sufficient quantities but not in excess. The use of large quantities of a chemical
often leads to a waste of time as it takes a longer time to dissolve. In addition, this may
lead to wrong results, e.g. if too much of lead iodide is used, its solubility in hot water will
not be observed.

• The following quantities should be used, unless otherwise specified:

Solid:

Enough to fill the **hemisphere** at the bottom of a **clean** and **dry** test-tube. The solid should be transferred by means of a **clean** and **dry spatula** into the bottom of the test-tube.

• Solution:

~1 cm depth (unless otherwise specified) of a clean test-tube.

• When testing the **solubility of precipitate in excess reagent**, pour out the excess precipitate until about 1 cm depth is left, before adding the excess reagent.



- 2. Place the filter paper in the funnel and wet it with a little deionised water.
- 3. Carefully pour the mixture from the test tube into the filter funnel.
- 4. Retain the filtrate and residue for further analysis if necessary.

3. Recording of Observations

- Indicate at which stage a change occurs by writing the observations alongside the tests performed. Record them **immediately after each test in ink**. Answers should include details such as:
 - **Colour** of salt or solution
 - Colour of precipitate (ppt) & its solubility in excess reagent
 - Colour change of solution
 - Colour of residue and filtrate after filtration
 - Colour of solid, before and after heating
 - Solubility of solid
 - Chemical test results for gas
- "Clear" is different from "Colourless". A clear solution may still be coloured. For example, aqueous Cu(NO)₂ is a clear blue solution.

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- "Effervescence" vs "Evolved":
 - "Effervescence" is for gases formed as bubbles during the reaction.
 - "Evolved" is when gases are produced but no bubbling is seen.
- Making deduction:
 - Make logical deductions based on the nature of the test reagents used.
 - Deduction is consequential ions that have been eliminated in the earlier tests need not be considered again in later deductions.
 - o Deduction should include the word 'present' or 'absent'.
 - Deductions should also be made for negative results, e.g. no ppt, no colour change, no gas evolved etc.
 - DO NOT write 'no change or no observation' for negative results.
- For a test with more than one step, align your observations and deductions with the corresponding steps in the test or state clearly at which stage the observations were made.

E.g.	<u>Test</u>	<u>Observation</u>	Deduction
	To 1 cm depth of FA 1 , add aqueous potassium manganate(VII)	purple KMnO₄ did not decolourise.	absence of reducing agent.
DF	To 1 cm depth of FA 1 , add aqueous ammonia,	white ppt observed	A <i>l</i> ³⁺ , Mg ²⁺ or Zn ²⁺ may be present
	then add aqueous ammonia in excess	ppt is soluble in excess NH ₃ .	Zn ²⁺ is present

• Acceptable short form: ppt (precipitate)

4. Appearance of Unknown

The appearance of the unknown in its solid form or in solution often provides a clue to its identity.

However, further chemical tests are needed to arrive at the final deductions of the unknown. This is because there are several possibilities for a given appearance or a mixture of unknowns could be given. E.g. a mixture of $FeCl_3$ and $CuCl_2$ solution appears green.

Appearance	Solid	Solution		
	hydrated Mn ²⁺ salts,	Mn ²⁺ (pale pink),		
Pink		MnO ₄ ⁻ (in very dilute solution),		
	hydrated Co ²⁺ salts	Co ²⁺		
	FeCl ₃ ,	Fe ³⁺ ,		
Brown	Mn(OH) ₃ , MnCO ₃ ,	I_2 in $I^-(I_3^-)$		
BIOWIT	anhydrous CuCl ₂ , CuBr ₂ ,	ζ		
	Ag ₂ O			
	Cu ₂ O, Cu ₂ Fe(CN) ₆ ,	Br ₂ in CCl ₄		
Reddish-brown	Fe ₂ O ₃ , Fe(OH) ₃ ,			
	Ag ₂ CrO ₄	2		
Orange	$K_2Cr_2O_7$,	$Cr_2O_7^{2-}$,		
		Br ₂ water		
	hydrated FeCl ₃ ,	$FeCl_3$, Fe^{3+} ,		
Yellow	BaCrO ₄ ,	CrO ₄ ^{2–} ,		
	K₄Fe(CN) ₆ ,	Fe(CN) ₆ ^{4–} ,		
	AgI	very dilute I₂ in I⁻		
	hydrated Fe ²⁺ salts,	Fe ²⁺ (very pale green),		
	hydrated CuCl ₂ , CuCO ₃ ,	Cu^{2+} (in concentrated solution),		
Green	Cr ₂ O ₃ ,	Cr^{3+} (bluish-green), $Cr(OH)_6^{3-}$,		
		$MnO_4^{2^-}$,		
	hydrated Ni ²⁺ salts, NiCO ₃			
Blue	nydrated Cu ²⁺ salts,	Cu^{2+} , $Cu(NH_3)4^{2+}$ (deep blue),		
		NI(NH ₃)6 ² ′		
5	Mao			
Black	NIC NIS			
	$\begin{bmatrix} N O, \\ O O \end{smallmatrix}$			
	$\Delta q_2 S_1 L_2$ (crystalline)			
	KMnO	MpQ_		
Purple		I_2 in CCL		
		Cr^{3+} (in concentrated solution)		
Red	K ₂ Fe(CN) ₆			
Grev with metallic	metals in powder form			
lustre				

5. Solubility of Salts in Water

Salts of	Soluble	Insoluble
NH4 ⁺ , Na ⁺ & K ⁺	all	-
NO ₃ ⁻	all	-
NO ₂ ⁻	all	Ag+
SO4 ²⁻	all	Ba ²⁺ , Ca ²⁺ & Ag ⁺
SO ₃ ²⁻	NH4 ⁺ , Na ⁺ , K ⁺	all
Cl [−] , Br [−] and I [−]	all	Ag+
CO ₃ ²⁻	NH4 ⁺ , Na ⁺ , K ⁺	all
OH⁻	NH4 ⁺ , Na ⁺ , K ⁺ (Ca ²⁺	all
	fairly soluble)	

6. Testing of lons

 The Qualitative Analysis Notes (see page 20–21) will be available during Practical assessment.

Note: while the observations are available in the Qualitative Notes (*Data Booklet*), you may be asked to explain the chemistry involved using **chemical equations** as well as relevant concepts from e.g. periodicity, electrochemistry and transition metal chemistry.

• List of possible cations & anions are:

Cations:	$\rm NH_4^+$	Mg ²⁺ Fe ²⁺	A <i>l</i> ³⁺ Fe ³⁺	Ca ²⁺ Cu ²⁺	Cr ³⁺ Zn ²⁺	Mn²+ Ba²+	H⁺	
Anions:	CO ₃ ^{2–}	NO₃⁻	NO ₂ -	SO4 ²⁻	SO3 ²⁻	Cl⁻	Br−	I-

Experiments may be conducted involving ions other than those listed above to draw conclusions of a general nature (e.g. whether they are oxidising or reducing, etc.)

1. Always check out the **physical appearance** of unknown solid/solution which often provides a clue to the detection of the ions present. E.g.: colour of a solid or solution. (Refer to section 4)

Do not perform other experiments except to test for a gas when appropriate.
 E.g. upon heating or when effervescence is observed.

6.1 Testing of cations [See experiment 12]

<u>Technique</u>

- 1. Add reagent [NH₃ (aq) / NaOH (aq) / Na₂CO₃ (aq)] **dropwise at first** to check for formation of precipitate. Observe and record colour of ppt.
- 2. For NaOH (aq) and NH₃ (aq), add these reagents **in excess** to check if ppt dissolves. Observe and record colour of solution formed. *(refer to Section 2.1 on how to check if ppt dissolves in excess)*

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6.1.1 Addition of aqueous sodium hydroxide

- precipitates insoluble hydroxides from solutions of metallic salts
- hydroxides of transition metals are usually coloured
- hydroxides such as Al(OH)₃, Cr(OH)₃ and Zn(OH)₂ are amphoteric and can dissolve in both acids and bases

• liberates weak volatile bases such as NH₃ on heating with ammonium salts

Reagent	Test for	Observation(s) and equation(s)
NaOH (aq)	Mg ²⁺	white ppt, Mg(OH) ₂ is formed; insoluble in excess NaOH (aq)
		$Mg^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mg(OH)_{2}(s)$
Test for: M ⁿ⁺		
Look for:	Mn ²⁺	off-white ppt, Mn(OH) ₂ is formed; rapidly turning brown on
1. ppt colour		contact with air, ppt is oxidised to Mn(OH) ₃
2. solubility of		<u>insoluble in excess</u> NaOH (aq)
ppt in excess		$Mn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mn(OH)_2 (s)$
NaOH(aq)		$4Mn(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Mn(OH)_3 (s)$
3. NH₃(g) when		2
asked to	Fe ²⁺	green ppt., Fe(OH) ₂ is formed; turning brown on contact with
warm mixture	0	air, ppt is oxidised to Fe(OH) ₃
		insoluble in excess NaOH (aq)
	\sim	$Fe^{2+}(aq) + 2OH^{-}(aq) \rightarrow Fe(OH)_2(s)$
	. 9	$4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)$
	Fe ³⁺	red-brown ppt, Fe(OH) ₃ is formed; insoluble in excess NaOH
		(aq)
~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~		$Fe^{3+}(aq) + 3OH^{-}(aq) \rightarrow Fe(OH)_{3}(s)$
	Cu ²⁺	pale-blue ppt, Cu(OH) ₂ is formed; insoluble in excess NaOH
		(aq)
		$Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2 (s)$
		pale blue ppt turned <u>black</u> on heating
		$Cu(OH)_2(s) \rightarrow CuO(s) + H_2O(l)$
	Cr ³⁺	grey-green ppt, Cr(OH) ₃ is formed; soluble in excess NaOH
		(aq) to form a <b>dark green solution</b> , [Cr(OH) ₆ ] ³⁻
		$Cr^{3+}(aq) + 3OH^{-}(aq) \rightarrow Cr(OH)_{3}(s)$
		$Cr(OH)_3(s) + 3OH^-(aq) \rightarrow [Cr(OH)_6]^{3-}(aq)$

NaOH (aq)	Zn ²⁺ , A <i>l</i> ³⁺	white ppt formed, soluble in excess NaOH (aq) to form a
		colourless solution
Test for: M ⁿ⁺		$Zn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Zn(OH)_2(s)$
		$Zn(OH)_2(s) + 2OH^-(aq) \rightarrow [Zn(OH)_4]^{2-}(aq)$
Look for:		
1. ppt colour		$Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Al(OH)_3(s)$
2. solubility of		$A_{l}(OH)_{3}(s) + OH^{-}(ag) \rightarrow [A_{l}(OH)_{4}]^{-}(ag)$
not in excess		
NaOH(an)	Ca ²⁺	white ppt is formed only when [Ca ²⁺ (ag)] is high
$3 NH_{1}(a)$ when	Uu	
S. WI3(g) WIEI	Ba ²⁺	<b>no nnt</b> is formed (if the reagents are pure)
warm mixture	Du	
	NH₄+	nungent gas evolved turns damp red litmus paper blue
	(warming	$\frac{\text{purigon gas}}{\text{NH}_{+}(\text{ag})} \rightarrow \text{OH}_{-}(\text{ag}) \rightarrow \text{NH}_{+}(\text{g}) + \text{H}_{-}(\text{g})$
	roguirod	$(aq) \rightarrow NI13(g) + 12O(l)$
	requirea)	

#### Re-precipitation of hydroxides

• With reference to **Section 6.1.1**, Al³⁺ (aq), Zn²⁺ (aq) and Cr³⁺ (aq) can be regenerated from their complexes by the dropwise addition of dilute nitric acid.



• Similarly, with reference to **Section 6.1.2 (later)**, Zn²⁺ (aq) and Cu²⁺ (aq) can be regenerated from their ammonia complexes by the dropwise addition of dilute nitric acid.



(Refer to Transition Metals Lecture Notes)

## 6.1.2 Addition of aqueous ammonia

- Like sodium hydroxide, aqueous ammonia also precipitates insoluble hydroxides from solutions of metallic salts.
- As aqueous ammonia is a weak alkali, it ionises partially in water:

$$NH_3 + H_2O \implies NH_4^+ + OH_4$$

For two solutions in which [NaOH] = [NH₃],  $[OH^{-}]_{NH_3} \le [OH^{-}]_{NaOH}$ .

Hence, slightly soluble hydroxides such as Ca(OH)₂ are not precipitated.

NH₃ (aq)	$M_{c}^{2+} = \Lambda I^{3+}$	
	Mg ^{-•} , At ^{-•}	<u>white ppt</u> is formed; <u>insoluble in excess</u> NH ₃ (aq) e.g. Mg ²⁺ (aq) + 2OH [−] (aq) → Mg(OH) ₂ (s)
Test for: M ⁿ⁺		
	Cr ³⁺	grey-green ppt, Cr(OH)₃ is formed; insoluble in excess
Look for:		NH ₃ (aq)
1. ppt colour		$Cr^{3+}(aq) + 3OH^{-}(aq) \rightarrow Cr(OH)_3(s)$
2. Solubility of	NA:==2+	off white ant Ma (OLI) is formed, could be turning brown a
NH ₂ (ad)		<u>contact with air</u> pot is ovidiced to Mp(OH), insoluble i
141 B(aq)		
		$\frac{excess}{Mn^{2+}(aq)} + 2OH^{-}(aq) \rightarrow Mn(OH)_{2}(s)$
		$4Mn(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Mn(OH)_3(s)$
	Fe ²⁺	green ppt,, Fe(OH) ₂ is formed; turning brown on contac
		with air, ppt is oxidised to Fe(OH) ₃ , insoluble in excess NH
		(aq)
		$Fe^{2+}(aq) + 2OH^{-}(aq) \rightarrow Fe(OH)_2(s)$
		$4Fe(OH)_2(s) + O_2(g) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s)$
	Fe ³⁺	red-brown ppt, Fe(OH)₃ is formed; insoluble in excess
	2	NH₃ (aq)
	25	Fe ³⁺ (aq) + 3OH [–] (aq) → Fe(OH) ₃ (s)
	Zn ²⁺	white ppt, Zn(OH) ₂ is formed; soluble in excess
	C V	NH ₃ (aq) to form a <u>colourless solution</u> , [Zn(NH ₃ ) ₄ ] ²⁺
		$Zn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Zn(OH)_2 (s)$
Ċ		Zn(OH)₂(s) + 4NH₃(aq) → [Zn(NH₃)₄]²+ (aq) + 2OH⁻(aq
	Cu ²⁺	blue ppt, Cu(OH) ₂ is formed; soluble in excess NH ₃ (aq) to
00		form a dark blue solution, [Cu(NH ₃ ) ₄ (H ₂ O) ₂ ] ²⁺
		Cu ²⁺ (aq) + 2OH [−] (aq) →Cu(OH) ₂ (s)
		$[Cu(H_2O)_6]^{2+}(aq) + 4NH_3(aq) \rightarrow [Cu(NH_3)_4(H_2O)_2]^{2+}(aq) +$
$\mathbf{v}$		$4H_2O(l)$
<u>2.</u>		

## 6.1.3 Addition of aqueous sodium carbonate

- Sodium carbonate solution is usually used to precipitate insoluble carbonates from solutions of metallic salts.
- In the presence of high charge density cations, effervescence of CO₂ will be observed (see * below).
- Due to hydrolysis, it can also act as base to produce hydroxide ions in solution.

$$CO_3^{2-} + H_2O \implies HCO_3^{-} + OH^{-}$$

Note: This table is not found in the Qualitative Analysis Notes provided.

Reagent	Test for	Observation(s) and equation(s)
Na ₂ CO ₃ (aq)	Mg ²⁺ , Ca ²⁺ , Ba ²⁺ , Zn ²⁺	$\frac{\text{white ppt}}{\text{e.g. Mg}^{2+}(\text{aq}) + \text{CO}_3^{2-}(\text{aq}) \rightarrow \text{MgCO}_3(\text{s})}$
Look for:	Mn ²⁺	$\frac{\text{off-white ppt.}}{\text{MnCO}_3 \text{ is formed}} \xrightarrow{\text{MnCO}_3} (s)$
2. CO₂(g) 3. NH₃(g) when asked to warm mixture	Fe ²⁺	<pre>green ppt,, FeCO₃ is formed Fe²⁺(aq) + CO₃²⁻(aq) → FeCO₃ (s) (Note that Fe²⁺ solutions are usually stabilised by acids. Adding bench reagents of Fe²⁺ and carbonate may give you effervescence af CO_due to reacting a paid and carbonate on the part of the second actions.</pre>
	Cu ²⁺	blue-green ppt, basic copper carbonate is formed. Effervescence of CO ₂ (g) Example when copper sulfate solution is used: $2 \text{ CuSO}_4 + 2 \text{ Na}_2\text{CO}_3 + \text{H}_2\text{O} \rightarrow \text{Cu}_2(\text{OH})_2\text{CO}_3 + 2 \text{ Na}_2\text{SO}_4 + \text{CO}_2$
	*Fe ³⁺	red-brown ppt, Fe(OH)₃ is formed. Effervescence of CO₂(g) (Need to observe carefully for tiny bubbles.)
	*A <i>l</i> ³⁺	white ppt, Al(OH) ₃ is formed. Effervescence of CO ₂ (g) (Need to observe carefully for tiny bubbles.)
4	*Cr ³⁺	grey-green ppt, $Cr(OH)_3$ is formed. Effervescence of $CO_2$ (g) (after some time)
<u> </u>	NH₄⁺ (warming may be required)	pungent gas evolved turns <u>damp red litmus paper blue</u> NH₄⁺(aq) + OH⁻(aq) → NH₃(aq) + H₂O( <i>l</i> )

*Why is CO₂ gas produced when Na₂CO₃ (aq) is added to a solution of  $Al^{3+}$  ions?

Due to the smaller cation size and a larger positive charge, and hence a higher charge density of  $Al^{3+}$  (high polarising power), it is able to polarise the O-H bonds of the neighbouring water molecules, thereby producing H⁺ in the solution.  $[Al(H_2O)_6]^{3+}(aq) \rightleftharpoons [Al(H_2O)_5(OH)]^{2+}(aq) + H^+(aq)$  $2H^+(aq) + CO_3^{2-}(aq) \rightarrow CO_2(g) + H_2O(l)$ 

Note:  $Al_2(CO_3)_3$  (s) is not produced. A similar explanation can be used for Fe³⁺ and Cr³⁺ ions.

#### 6.1.4 Additional tests for cations

	Rea	actions that you must know
Reagent	Test for	Observation(s) and equation(s)
H₂SO₄ (aq) Test for: Ba²⁺ Look for: ppt	Ba ²⁺	white ppt is formed. e.g. $Ba^{2+} + SO_4^{2-} \rightarrow BaSO_4$ (s)
HCl (aq) Test for: Ag ⁺ Look for: ppt	Ag⁺	white pptis formed. $Ag^+ + Cl^- \rightarrow AgCl$ (s)Note: check for solubility of ppt in dilute NH3.
Concentrated HC <i>l</i> Look for: solution colour change	Cu ²⁺	blue solution turns <u>yellowish-green</u> (mixture of 2 complexes) $[Cu(H_2O)_6]^{2+} + 4Cl^- \rightarrow [CuCl_4]^{2-} + 6H_2O$ blue yellow
KI (aq), aqueous potassium iodide	Cu ²⁺	$\begin{array}{c} \underline{\text{cream ppt}} \text{ in } \underline{\text{brown solution}}\\ 2Cu^{2+} + 4I^- \rightarrow 2CuI \text{ (s)} + I_2\\ I_2 + I^- & \longrightarrow I_3^- \text{ (brown solution)} \end{array}$
Look for: ppt; solution colour change when Na₂S₂O₃ is added (for I⁻)		$\begin{array}{l} \displaystyle                                   $
	Good to k	now but not required to memorise
Reagent C	Test for	Observation(s) and equation(s)
K ₄ Fe(CN) ₆ (aq),	Cu ²⁺	reddish-brown ppt is formed.
hexacyanoferrate(II)	Fe ²⁺	pale blue ppt is formed.
Test for: Cu ²⁺ , Fe ²⁺ ,	Fe ³⁺	prussian blue ppt is formed.
Fe st , Zn <del>r</del> Look for: ppt colour	Zn ²⁺	white ppt is formed.
NH₄SCN(aq), aqueous ammonium	Cu ²⁺	black ppt is formed.
uniocyanale,	Fe ²⁺	colourless solution (solution may turn pink or orange du

This table is not found in the Qualitative Analysis Notes provided.

Test for: Cu²⁺, Fe²⁺, Fe³⁺ to slight oxidation of  $Fe^{2+}$  to  $Fe^{3+}$ ) <u>deep red colouration</u>.  $[Fe(SCN)(H_2O)_5]^{2+}$  complex is Fe³⁺ Look for: ppt colour or solution colour formed.

# 6.2 Testing of anions [See experiment 12]

Reagent	Test for	Observation(s) and equation(s)
Aluminium foil (Al) heated with NaOH (aq)	<b>NO</b> ₃ ⁻ or <b>NO</b> ₂ ⁻ (in absence of $NH_4^+$ )	pungent gas evolved turns damp red litmus paper blue NO ₂ ⁻ + OH ⁻ + 2A/ + 5H ₂ O → NH ₃ + 2A/(OH) ₄ ⁻
Test for: NO ₃ ⁻ , NO ₂ ⁻ Look for: NH ₃ (g)		$3NO_3^- + 5OH^- + 8Al + 18H_2O \rightarrow 3NH_3 + 8Al(OH)_4^-$
		If NH ₄ ⁺ ions are present, boil the solution with NaOH (aq) until no more NH ₃ (g) is evolved before adding Al foil. This prevents false positive test for NO $_{2}^{-}$ NO $_{2}^{-}$
dilute H ₂ SO ₄ or dilute HNO ₃ or dilute HC <i>l</i>	CO ₃ ^{2–} , HCO ₃ –	$\begin{array}{l} \hline \textbf{gas} \text{ evolved gives a } \underline{\textbf{white ppt}} \text{ with } \underline{\textbf{limewater}} \\ \hline \textbf{CO}_3^{2-} + 2\textbf{H}^+ \rightarrow \textbf{CO}_2 + \textbf{H}_2\textbf{O} \\ \hline \textbf{HCO}_3^{-} + \textbf{H}^+ \rightarrow \textbf{CO}_2 + \textbf{H}_2\textbf{O} \end{array}$
Test for: CO₃²-, SO₃²-, NO₂ ⁻ , HCO₃ ⁻	<b>SO</b> ₃ ^{2–} (not tested)	pungent gas evolved turns aqueous <u>acidified</u> <u>KMnO₄ from purple to colourless</u> $SO_3^{2-} + 2H^+ \rightarrow SO_2 + H_2O$
Look for: CO ₂ (g), SO ₂ (g) and NO ₂ (g)	NO ₂ -	$\begin{array}{l} \underline{\textbf{pungent brown gas}} \text{ is evolved} \\ NO_2^- + H^+ \rightarrow HNO_2 \\ 2HNO_2 \rightarrow NO_2 + NO + H_2O \\ NO + \frac{1}{2}O_2 \rightarrow NO_2 \end{array}$
	CrO ₄ ^{2–}	$\frac{\text{yellow solution turns orange}}{2\text{CrO}_4^{2-} + 2\text{H}^+ \rightarrow \text{Cr}_2\text{O}_7^{2-} + \text{H}_2\text{O}}$
AgNO ₃ (aq), followed by NH ₃ (aq) <i>Test for: X</i> ⁻	CI-	white ppt, AgCl is seen, soluble in NH ₃ (aq) Ag ⁺ + Cl ⁻ → AgCl (s) Ag ⁺ + NH ₃ $\implies$ [Ag(NH ₃ ) ₂ ] ⁺ (aq)
Look for: ppt colour; solubility of ppt in NH ₃ (aq)	Br	pale cream ppt, AgBr is seen, partially soluble in NH₃ (aq) Ag⁺ + Br⁻ → AgBr (s)
6	1-	<u><b>yellow ppt</b></u> , AgI is seen, insoluble in NH ₃ (aq) Ag ⁺ + I ⁻ → AgI (s)
Ba(NO ₃ ) ₂ (aq) <b>or</b> BaCl ₂ (aq), followed by acid (HNO ₃ or HC <i>l</i> )	SO4 ²⁻	white ppt, BaSO ₄ is formed, insoluble in dilute HCl or HNO ₃ Ba ²⁺ + SO ₄ ^{2−} → BaSO ₄ (s)
Test for: SO₃²-, SO₄²- Look for: ppt; solubility of ppt in acid	<b>SO</b> ₃ ^{2–} (not tested)	$\begin{array}{l} \underline{\textbf{white ppt}}, \ BaSO_3 \ \text{is formed}, \ \underline{\textbf{soluble}} \ \text{in dilute HC}{\it l} \ \text{or} \\ \text{HNO}_3. \ SO_2 \ \text{gas evolved}. \\ Ba^{2+} + SO_3^{2-} \rightarrow \ BaSO_3 \ (s) \\ BaSO_3 + 2H^+ \rightarrow Ba^{2+} + SO_2 \ (g) + H_2O \end{array}$
	CO3 ²⁻	$\begin{array}{l} \hline \textbf{white ppt}, \ BaCO_3 \ is \ formed, \ \textbf{soluble} \ in \ dilute \ HCl \ or \\ HNO_3. \ CO_2 \ gas \ evolved. \\ Ba^{2+} + CO_3^{2-} \rightarrow \ BaCO_3 \ (s) \\ BaCO_3 + 2H^+ \rightarrow Ba^{2+} + CO_2 \ (g) + H_2O \end{array}$

KMnO ₄ (acidified with <u>equal</u> <u>volume</u> of dil. H ₂ SO ₄ ) Test for: Reducing agents	reducing agent (e.g. I [−] ) (warming may be required)	decolourisation of purple KMnO ₄ solution note down any gas/ppt/solution colour change
Look for: ppt; gas; solution colour change		

#### **Re-precipitation of silver halides**

x by contractions of the second secon Silver chloride can be re-precipitated from the diammine silver(I) complex by the dropwise

# 7. Separation of lons

This section is concerned with how to separate ions in a given mixture of ions. Separation of ions is based on the <u>different solubilities of compounds in different reagents</u>. You will have to be familiar with the different reactions of cations and anions with the common reagents described in **Section 6**.

#### Example (given a mixture of Zn²⁺ and Cu²⁺)

 $Zn^{2+}$  reacts with NaOH to form  $Zn(OH)_2$  (white ppt) which dissolves in excess NaOH to give a soluble complex  $Zn(OH)_4^{2-}$  (colourless solution). On the other hand,  $Cu^{2+}$  reacts with NaOH to form  $Cu(OH)_2$  (pale blue ppt) which is insoluble in excess NaOH.

Since the two hydroxides formed dissolve in excess NaOH to different extent, by filtering the insoluble hydroxide, we can separate the ions from each other.

Recovery of the dissolved Zn²⁺ ions can be effected by adding another reagent to reprecipitate the hydroxide.



#### **Reactions of Common Oxidising and Reducing Agents** 8.

#### 8.1 **Oxidising Agents**

Oxidising	Reduction of Oxidising Agent	Observation	
Agent			
	$MnO_4^- \rightarrow Mn^{2+}$ (acidic medium)	purple solution decolourised	
MnO ₄ -	$MnO_4^- \rightarrow MnO_2(s)$ (alkaline medium)	purple solution decolourised and	
		brown ppt forms	
Cr ₂ O ₇ ²⁻	$Cr_2O_7^{2-} \rightarrow Cr^{3+}$	orange to green	
H-O-	$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O$ (acidic medium)		
$\Pi_2 \cup_2$	$H_2O_2 + 2e^- \rightarrow 2OH^-$ (alkaline medium)		
Br ₂ (aq)	$Br_2 \rightarrow Br^-$	orange solution decolourised	
I₂ (in I⁻)	$I_2 \rightarrow I^-$	brown solution decolourised	
8.2 Reducing	g Agents	, P	

#### 8.2 **Reducing Agents**

Reducing Agent	Oxidation of Reducing Agent	Observation
I-	$I^- \rightarrow I_2$	brown solution forms
Fe ²⁺	$Fe^{2+} \rightarrow Fe^{3+}$	pale green to yellow
SO3 ²⁻	$SO_3^{2-} \rightarrow SO_4^{2-}$	
NO ₂ ⁻	$NO_2^- \rightarrow NO_3^-$	
H ₂ O ₂	$H_2O_2 \rightarrow O_2$	

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# 9. Test for Gases

## [See experiment 13]

- Gases or vapours liberated during **dry heating** or by the **addition of reagents such as acids or alkalis** should first be detected according to their colour, odour and action on damp litmus paper. They should then be identified using chemical tests.
- Quantity of substance to use, unless otherwise specified:
  - o Solid: at least 2 to 3 spatula OR enough to cover the bottom hemisphere of boiling tube
  - $\circ$  Amount of limewater to test for CO₂: about 1/3 length of a test-tube
- Prepare the necessary reagent/ apparatus beforehand and conduct the test as follows:

Gas	Technique	Observation
ammonia, NH ₃ (pungent smell)	Hold <b>damp red litmus</b> paper at the <b>mouth</b> of the test-tube, without touching the sides of the test-tube	pungent gas evolved turns damp red litmus paper blue
carbon dioxide, CO ₂	Method 1: Deliver gas via a <b>delivery tube</b> into another test-tube containing <b>limewater</b> [Ca(OH) ₂ (aq)] <u>Method 2</u> : Use a dropper to draw up some gas and bubble into another test-tube containing <b>limewater</b>	gas evolved gives a <u>white</u> <u>ppt</u> with <u>limewater</u> (ppt dissolves in excess CO ₂ )
sulfur dioxide, SO ₂ (smell of burning sulfur) (not tested)	<u>Method 1</u> : Deliver gas via a <b>delivery tube</b> into another test-tube containing <b>equal</b> <b>volumes of KMnO</b> ₄ ( <b>aq</b> ) and <b>dilute</b> H ₂ SO ₄ <u>Method 2</u> : Add a <b>few drops of dilute</b> H ₂ SO ₄ followed by a <b>few drops of KMnO</b> ₄ onto a <b>filter paper strip</b> and hold the filter paper strip at the mouth of the test-tube, without touching the sides of the test-tube	pungent gas evolved turns aqueous <u>acidified KMnO₄</u> from purple to colourless
oxygen, O ₂	Use a <b>glowing</b> splint	gas evolved <u>relights</u> glowing splint
chlorine, Cl ₂ (pale yellowish- green; pungent smell; colour more easily seen when dissolved in organic solvent)	Hold <b>damp blue</b> litmus paper at the <b>mouth</b> of the test-tube, without touching the sides of the test-tube	pungent gas evolved turns damp blue litmus paper red then bleaches it
hydrogen, H ₂	Use <b>lighted</b> splint	gas evolved gives a 'pop' sound with a lighted splint

# 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

In organic reactions and other reactions, a test for halogens is sometimes carried out as follows.

Test	Observation for positive test
To a test-tube containing 1 cm ³ of the sample, add a few drops of organic solvent (e.g. hexane) and shake.	The organic layer turns from colourless to <u>(colour of halogen)</u> . organic layer

	$\mathcal{A}$
2AM	
SE	
SOL	
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Y	

# 10. Heating

#### 10.1 Technique

- Close the air hole of the bunsen burner when lighting the flame. Once the flame is on, adjust the air hole to give a non–luminous flame. Normal heating is usually done with a bunsen flame in which the air hole of the burner is half opened.
- Get ready the materials needed to test for gas liberated before carrying out the heating.

_		
	When heating a <b>solid</b> ,	When heating a <b>solution</b> , spurting of the
	<ul> <li>Use a <u>clean</u> and <u>dry</u> test tube / boiling tube.</li> </ul>	solution can be prevented by:
	✤ Hold the tube in an <u>almost horizontal</u>	Apply a small flame near the surface/
	position.	meniscus of the solution.
	Apply heat <u>gently initially</u> (to remove any	The test tube should not be held in a
	water of crystallisation present) and then	fixed position all the time but should be
	strongly until no further change takes	then and shaken dently
		<u>unon</u> and on anon gonity.
	✤ Be careful not to heat till the solid	
I	decomposes.	



- Test for gas (if any) liberated. This would indicate the presence of a certain anion. The following sequence can be used:
  - 1. use damp red and blue litmus papers. [to test for acidic gases or  $NH_3$  (g)]
  - **2.** bubble gas through another test-tube containing limewater (aqueous calcium hydroxide) [to test for  $CO_2$  (g)]
    - use glowing splint [to test for O₂ (g)]

#### **10.2** Observations and Deductions

• Most solids undergo changes when heated. These changes often provide a clue to the cation and/or anion present in the solid.

presence of	observation of residue	
Zn ²⁺	yellow when hot, white when cold	
Cu ²⁺ , Mn ²⁺ , Ni ²⁺	black residue from blue/ green substances	
Fe ²⁺ , Fe ³⁺	brown	
Cr ³⁺	green	<
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presence of	possible gas liberated
CO ₃ ^{2–} , HCO ₃ –	CO ₂
NO ₃ -	NO ₂
NO₂ [−]	NO ₂
$SO_4^{2-}$ , $SO_3^{2-}$	
NH4 ⁺	NH ₃
HCO ₃ -,	
compounds with water of crystallisation,	H ₂ O
hydroxides of metals except KOH and NaOH	
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# **10. Qualitative Analysis Notes** [ppt. = precipitate]

# (a) Reactions of aqueous cations

cation	reaction with	
callon	NaOH(aq)	NH₃(aq)
aluminium,	white ppt.	white ppt.
Al ³⁺ (aq)	soluble in excess	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), Cr³+(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), 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), Mn²⁺(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, Zp ²⁺ (2g)	white ppt. soluble in excess	white ppt. soluble in excess

# (b) Reactions of anions

ion	reaction
carbonate, CO ₃ ²⁻	CO ₂ liberated by dilute acids
chloride, C <i>l</i> ⁻(aq)	gives white ppt. with Ag⁺(aq) (soluble in NH₃(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 A <i>l</i> foil
nitrite, NO ₂ -(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> 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 dilute strong acids)
(c) Tests for gases	

# (c) Tests for gases

gas	test and test result
ammonia, NH₃	turns damp red litmus paper blue
carbon dioxide, CO2	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

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