#### Table 15 - Salt

#### Subject content:

- (a) describe the techniques used in the preparation, separation and purification of salts as examples of some of the techniques (methods for preparation should include precipitation and titration together with reactions of acids with metals, insoluble bases and insoluble carbonates)
- (b) describe the general rules of solubility for common salts to include nitrates, chlorides (including silver and lead), sulfates (including barium, calcium and lead), carbonates, hydroxides, salts of Group I cations and ammonium salts
- (c) suggest a method of preparing a given salt from suitable starting materials, given appropriate information

#### 15.1 Salts and Solubility

**Salt**: ionic compound that contains a **cation** (+) and **anion** (–)

• Formed when metallic / ammonium cation replaces H<sup>+</sup> ions of acid

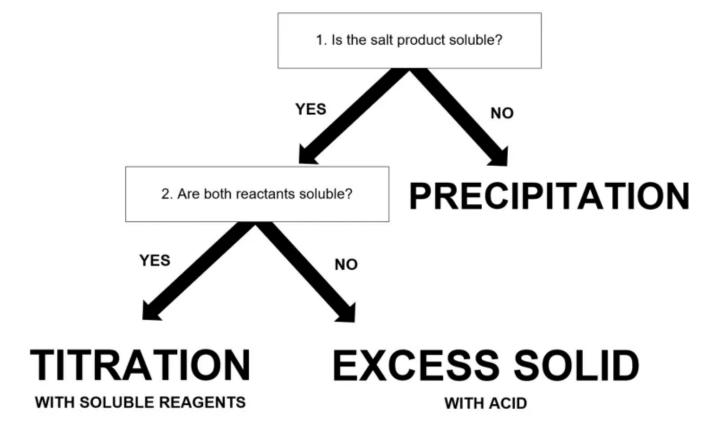
• Preparation reactions:

Reactants	Products
acid + metal	salt + H <sub>2</sub>
acid + carbonate	salt + water + CO <sub>2</sub>
acid + base	salt + water
acid + alkali	salt + water

Solubility of salts:

Type of salt	Soluble	Insoluble
Grp I (Na <sup>+</sup> , K <sup>+</sup> )	All	
Ammonium (NH <sub>4</sub> <sup>+</sup> )	All	
Nitrates (NO <sub>3</sub> -)	All	
Chlorides (Cl <sup>-</sup> )	All others	• Ag • Pb
Sulfates (SO <sub>4</sub> <sup>2-</sup> )	All others	<ul><li>Ag</li><li>Pb</li><li>Ba</li><li>Ca</li></ul>
lodides (I <sup>-</sup> )	All others	<ul><li>Ag</li><li>Pb</li></ul>
Carbonates (CO <sub>3</sub> <sup>2-</sup> )	<ul><li> Grp I metals</li><li> Ammonium</li></ul>	All others
Hydroxides (OH <sup>-</sup> )	<ul><li> Grp I metals</li><li> Ammonium</li></ul>	All others
Oxides (O <sup>2-</sup> )	<ul><li> Grp I metals</li><li> Ammonium</li></ul>	All others

### 15.2 Salt Preparation



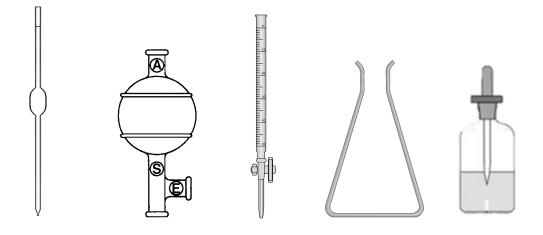
Salt preparation:

Method	Explanation	Procedure	Examples
1. Titration	<ul> <li>Acid + alkali</li> <li>Acid + soluble carbonate</li> </ul>	<ul> <li>Preparation</li> <li>1) Pour (acid) into burette</li> <li>2) Pipette 25.0 cm³ (alkali) into conical flask</li> <li>3) Add a few drops of screened methyl orange (indicator) into conical flask</li> <li>4) Add (acid) from burette into conical flask until screened methyl orange → grey / colourless</li> <li>5) Record volume of (acid) used</li> <li>6) Repeat experiment without indicator, using recorded volume of (acid)</li> <li>Obtaining (crystallisation)</li> <li>1) Pour (salt solution) into evaporating dish. Heat → saturated</li> <li>2) Cool solution → crystals form</li> <li>3) Filter mixture → obtain residue (salt)</li> <li>4) Dry (salt) between sheets of filter paper</li> </ul>	KOH + HNO <sub>3</sub> → KNO <sub>3</sub> + H <sub>2</sub> O • $\sqrt{\text{K}_2\text{CO}_3}$ • X K (too reactive)
2. Acid + insoluble substance	<ul> <li>Acid + excess insoluble base</li> <li>Acid + excess metal</li> <li>Acid + excess insoluble carbonate</li> </ul>	<ul> <li>Preparation</li> <li>1) Pour (acid) into beaker</li> <li>2) Add (solid) a little at a time with stirring, into warm acid until (solid) is in excess (ensure all the acid is used up)</li> <li>Obtaining (filtration + crystallisation)</li> <li>1) Filter to remove excess (solid). Collect filtrate (salt solution)</li> <li>2) Pour (salt solution) into evaporating dish. Heat → saturated</li> <li>3) Cool solution → crystals form</li> <li>4) Filter mixture → obtain residue (salt)</li> <li>5) Dry (salt) between sheets of filter paper</li> </ul>	H <sub>2</sub> SO <sub>4</sub> + CuO → CuSO <sub>4</sub> + H <sub>2</sub> O  • √ CuO, Cu(OH) <sub>2</sub> , CuCO <sub>3</sub> • X Cu (unreactive)

3. Precipitation	Solution containing cation of salt + solution containing anion of salt	Preparation 1) Pour excess (aq sol. 1) into (aq sol. 2)  Obtaining (filtration) 1) Filter mixture 2) Wash the residue (precipitate) with distilled water  → remove excess (aq sol. 1) 3) Dry (precipitate) between sheets of filter paper	AgNO <sub>3</sub> + NaC <i>I</i> → AgC <i>I</i> + NaNO <sub>3</sub> • must use 2 aqueous solutions • not typical acid reaction (does not involve H <sup>+</sup> ions) • Ag <sup>+</sup> (aq) + C <i>I</i> <sup>-</sup> (aq) → AgC <i>I</i> (s)
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# 15.3 Titration (Practical) Apparatus and chemicals used:

Apparatus and chemical Function				
1. Pipette	Accurately measure fixed volume of liquid (25.0 cm³)			
2. Pipette filler	Extract solution into	Extract solution into the pipette		
3. Burette	<ul> <li>Accurately measurevolume of liquid to nearest 0.05 cm<sup>3</sup></li> <li>Deliver different volumes of solutions</li> </ul>			
4. Conical flask	•	<ul> <li>Contain aqueous reactants for reaction</li> <li>Broad base + small mouth → better swirling + reduce spillage</li> </ul>		
	Determine end-point (complete reaction) → colour change			
	la dia atau	Colour	Colour	
	Indicator	(effective pH range)	Acidic	Alkaline
	Methyl orange	Orange (3 – 5)	Red	Yellow
5. Indicator	Screened methyl orange	Grey (3 – 5)	Violet	Green
	Thymolphthalein	Blue (8 – 10)	Colourless	Blue
	Acid-base titration  No colour change –  No indicator is requ	•		ourless



Cleaning apparatus

Preparation	Purpose
Rinse with tap water	Remove water soluble containments
2. Rinse with distilled / deionised water	Remove unwanted ions
<ol> <li>Rinse with alkali (burette + pipette) →  use clean funnel to direct solution  without spillage</li> </ol>	Remove any water droplets left to prevent dilution (Conical flask not rinsed with alkali → do not introduce extra solution into it)

#### Filling burette with alkali:

- 1. Place retort stand on stool before filling burette with alkali
- 2. Ensure tap is closed
- 3. Using funnel, fill burette until level of alkali is slightly above 0 mark
- 4. Remove funnel from burette before starting titration
  - → no extra alkali added to burette during titration
- 5. Open tap  $\rightarrow$  allow some alkali to run out to make sure that there is no trapped air
- 6. Ensure burette is clamped vertically upright
- 7. Read level of alkali in burette (eye: same level as bottom of meniscus)
- 8. Record initial burette reading
- 9. Move retort stand with burette back to bench

#### Using pipette to measure & transfer acid into conical flask:

- 1. Pipette filler: hold (A) and compress
- 2. hold pipette near the top, insert gently into pipette filler
- 3. place tip of pipette into beaker containing acid → below surface of solution
- 4. Pipette filler: press and hold (S) to fill pipette → slightly above 25.0 cm³ mark
- 5. Pipette filler: press and hold (E) to release some acid as waste → meniscus of solution reaches 25.0 cm³ mark (eye level)
- 6. remove pipette filler  $\rightarrow$  allow acid to run into conical flask

#### Adding indicator into conical flask:

- 1. Add a few drops  $(2 \sim 3)$  of indicator (e.g. screened methyl orange)
- 2. Swirl conical flask gently → mix indicator & solution
- 3. Place a white tile underneath conical flask on burette

#### Carrying out titration:

- 1. Add alkali in burette to acid in conical flask
- 2. Swirl conical flask gently → contents mixed well
- 3. As alkali is added to conical flask, colour of solution starts to change
- 4. Add alkali **drop by drop** while swirling conical flask gently
- 5. stop titration when solution has just turned from green  $\rightarrow$  grey (subject to indicator)
- 6. take final burette reading
- 7. 1st titration → estimated volume of acid for complete reaction

#### Recordings:

Experiment no.	1	2	3
Final burette reading / cm <sup>3</sup>	29.50	29.10	32.00
Initial burette reading / cm <sup>3</sup>	3.10	4.40	7.20
Volume of (alkali) / cm <sup>3</sup>	26.40	24.70	24.80
Best titration results (√)		√	√

Consistent readings: ± 0.10 cm<sup>3</sup> of each other

#### 15.4 Qualitative Analysis

Qualitative analysis: process of identifying cations and anions

#### Identifying cations

Reagents used to identify cations

- 1. Sodium hydroxide solution (NaOH)
- 2. Aqueous ammonia (NH<sub>3</sub>)
- 3. All cations (except Na<sup>+</sup>, K<sup>+</sup>, NH<sub>4</sub><sup>+</sup>) give precipitates with alkalis

#### Identify cations by noting:

- Colour of precipitate produced
- Solubility of precipitate in excess of reagent
- Whether ammonia gas is liberated on NaOH solution

Cation	Sodium hydroxide solution (NaOH)		Aqueous ammonia (NH <sub>3</sub> )	
Cation	Add few drops	Add excess	Add few drops	Add excess
Zn <sup>2+</sup>		ppt. <u>dissolve</u> → colourless sol		ppt. <u>dissolve</u> → colourless sol
Al <sup>3+</sup>	white ppt.	ppt. <u>dissolve</u> → colourless sol	white ppt.	ppt. <u>insoluble</u>
Pb <sup>2+</sup>	<u>winto</u> ppt.	ppt. <u>dissolve</u> → colourless sol		ppt. <u>insoluble</u>
Ca <sup>2+</sup>		ppt. <u>insoluble</u>	<u>no</u> ppt.	no ppt.
Cu <sup>2+</sup>	<u>light</u> <u>blue</u> ppt.	ppt. <u>insoluble</u>	<u>light</u> <u>blue</u> ppt.	ppt. <u>dissolve</u> → deep blue sol
Fe <sup>2+</sup>	green ppt.	ppt. <u>insoluble</u>	<u>green</u> ppt.	ppt. <u>insoluble</u>
Fe <sup>3+</sup>	reddish-brown ppt.	ppt. <u>insoluble</u>	reddish-brown ppt.	ppt. <u>insoluble</u>
NH <sub>4</sub> <sup>+</sup>	no ppt. (ammonia gas given off on heating)	no change		

Precipitate formed: hydroxide of metal ion

Some precipitates dissolve in excess NaOH / NH<sub>3</sub>(aq) – formation of soluble compounds in water

## Identifying anions Common anions

Anion	Test	Observation (+ve)
CO <sub>3</sub> <sup>2-</sup>	Add HC/     Pass gas given off into limewater	Effervescence (CO <sub>2</sub> given off)
NO <sub>3</sub> -	<ul> <li>Add NaOH</li> <li>Add aluminium foil, warm mixture</li> <li>Test gas given off with moist red litmus paper</li> </ul>	Effervescence (NH <sub>3</sub> given off)
SO <sub>4</sub> <sup>2-</sup>	<ul> <li>Add HNO<sub>3</sub></li> <li>Add Ba(NO<sub>3</sub>)<sub>2</sub></li> </ul>	White precipitate (BaSO <sub>4</sub> )
C/ <sup>-</sup>	<ul> <li>Add HNO<sub>3</sub></li> <li>Add AgNO<sub>3</sub></li> </ul>	White precipitate (AgC/)
I-	<ul> <li>Add HNO<sub>3</sub></li> <li>Add AgNO<sub>3</sub></li> </ul>	Yellow precipitate (AgI)

Identifying gases
Gas liberated when unknown salt is tested

Ga	S	Colour & odour	Test	Observation
o d	Hydrogen (H <sub>2</sub> )			Lighted splint extinguished with 'pop' sound
o u	Oxygen (O <sub>2</sub> )	colourless odourless	Insert glowing splint into test tube	Glowing splint is rekindled
rl e s	Carbon dioxide (CO <sub>2</sub> )	colourless odourless	Bubble gas through limewater	White precipitate formed Precipitate dissolves upon further bubbling
p u	Chlorine (Cl <sub>2</sub> )	greenish-yellow pungent	Place moist blue litmus paper at mouth of test tube	Moist blue litmus paper turns red, and is then bleached
n g e n	Sulfur dioxide (SO <sub>2</sub> )	colourless pungent	Place filter paper soaked with acidified potassium manganate(VII) at mouth of test tube	Acidified potassium manganate(VII) turns from purple to colourless
t	Ammonia (NH <sub>3</sub> )	colourless pungent	Place moist red litmus paper at mouth of test tube	Moist red litmus paper turns blue

#### **Tests for water**

Heat hydrated salt → water condenses near top of test tube

Test	Procedure	Observation
Cobalt(II) chloride paper	Heat sample in test tube. Place test paper at mouth of test tube.	blue → pink
2. Anhydrous copper(II) sulfate	Add few drops of sample	white → blue

#### **Typical questions**

#### **Multiple choice questions**

- 1 Which ions, when combined with chloride ions, CI-, forms a precipitate in water?
  - **A** Fe<sup>2+</sup>
  - **B** Pb<sup>2+</sup>
  - C Mg<sup>2+</sup>
  - **D** Na⁺
- 2 The following methods for preparing salts are given.
  - 1 Reaction of an acid with an insoluble base / metal / carbonate
  - 2 Reaction between two solutions to form an insoluble salt
  - 3 Reaction of an acid with another soluble reactant such as an alkali
  - 4 Sodium sulfate, Na2SO4 (aq), can be prepared by:
  - A 1 only
  - **B** 1 and 3
  - **C** 2 and 3
  - **D** 1, 2 and 3

#### **Qualitative analysis**

**1** Ammonia solution is added in excess to a solution containing lead, calcium, zinc, iron(III) and copper(II) cations. Which one of the following observations is correct?

	Cation	Observation	
Α	Iron(III)	Reddish brown precipitate, insoluble in excess	
В	Calcium	White precipitate, soluble in excess	
С	Zinc	White precipitate, insoluble in excess	
D	Lead	White precipitate, soluble in exess	

2	A gas	has	the	following	properties:
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- It is soluble in water
- It is colourless
- It turns moist red litmus paper blue

This gas could be:

- E carbon dioxide
- F hydrogen
- **G** ammonia
- **H** oxygen
- **3** Which one of the following tests can be used to distinguish between potassium iodide and potassium chloride?
  - **A** Heat strongly
  - **B** Use litmus paper
  - C Add aqueous ammonia
  - **D** Add dilute nitric acid, followed by aqueous silver nitrate
- **4** When aqueous sodium hydroxide is added to solution W, a green precipitate is produced. The green precipitate turns brown after a while. Which ion is present in solution W?
  - **A** Al<sup>3+</sup>
  - **B** Cu<sup>2+</sup>
  - **C** Fe<sup>2+</sup>
  - **D** Fe<sup>3+</sup>
- **5** A solution of an unknown substance produces a white precipitate with acidified silver nitrate solution. When added to aqueous ammonia, a white precipitate that dissolves in excess alkali is produced. What is the unknown substance?
  - A zinc chloride
  - B calcium iodide
  - C lead(II) chloride
  - **D** aluminium iodide

- 6 A sample of an unknown powder is added to dilute hydrochloric acid. Effervescence is observed and a colourless and odourless gas is produced. It is neutral to moist litmus paper but extinguished a burning splint with a 'pop' sound. When aqueous ammonia is added, a white precipitate is formed which is insoluble in excess alkali. When acidified potassium iodide is reacted with the powder, a yellow precipitate is formed. What is the unknown powder?
  - A zinc
  - **B** lead
  - C calcium
  - **D** aluminium
- **7** When a certain green powder is heated, it produces a black powder X and a gas Y. Gas Y gives a white precipitate in limewater.

Black powder X is added to dilute sulfuric acid and a blue solution is formed after filtering. This solution is heated to remove most of the solvent. Upon cooling, blue crystals Z are formed. Upon addition of concentrated sulfuric acid, they turn white.

(a) Suggest what the green powder was.

copper(II) carbonate

- **(b)** Identify
  - (i) black powder X

copper(II) oxide

(ii) gas Y

carbon dioxide

(iii) blue crystals Z

copper(II) sulfate

**(c)** Explain why the blue crystals turn white.

They lose their water of crystallisation and become anhydrous. Copper(II) sulfate is used to test for the presence of water vapour.

- 8 An alloy which contains two metals was divided into two samples.
  - When the first sample was added to excess dilute hydrochloric acid, a pink solid and colourless solution were formed. When excess aqueous ammonia was added to the colourless solution, a white precipitate was formed.
  - The second sample was heated with excess nitric acid to form a blue solution. In the presence of excess sodium hydroxide solution, a blue precipitate was observed.
  - (a) Name the pink solid and blue solution.

The pink solid is copper and the blue solution is a mixture of copper(II) nitrate and aluminium nitrate solution.

The first test shows the presence of aluminium. Aluminium can react with dilute acid to form colourless solution and hydrogen gas. The pink solid is a metal that does not react with dilute hydrochloric acid. The second test shows that the pink solid is copper. Copper does not react with dilute acids but can be oxidised by nitric acid.

**(b)** Explain why zinc cannot be present in the alloy.

If zinc were present, it would form a white precipitate with aqueous ammonia that would react in the presence of excess alkali.

(c) The colourless solution from the first sample was reacted with acidified potassium iodide. No reaction was observed. What are the two metals present in the alloy?

Aluminium and copper

(d) Name the white precipitate formed in the first sample. Write the equation for the reaction leading to its formation.

The white precipitate is aluminium hydroxide.

 $Al^{3+} + 3 OH^{-} \rightarrow Al(OH)_{3}$