

QUALITATIVE ANALYSIS

SAMPLE NO:

AIM: TO ANALYSE THE GIVEN INORGANIC SALT QUALITATIVELY AND DETECT ONE CATION AND ONE ANION.		
EXPERIMENT	OBSERVATION	INFERENCE
1. PHYSICAL CHARACTERISTICS a) COLOUR	Blue/Bluish-green.	$\text{Cu}^{2+}/\text{Ni}^{2+}$
	Green	Ni^{2+}
	Brown/Blackish.	Fe^{3+}
	White	$\text{Cu}^{2+}, \text{Ni}^{2+}, \text{Fe}^{3+}, \text{Co}^{2+}$ are absent.
b) ODOUR	Ammonical	NH_4^+ may be.
	Vinegar like smell.	CH_3COO^- may be.
2. DRY-HEATING TEST Heat a pinch of sample in a clean and dry test tube.	White sublimate.	NH_4^+ may be.
	Brown gas.	$\text{NO}_2 + \text{NO}_3^-$
	Residue turns black.	$\text{Cu}^{2+}/\text{Ni}^{2+}/\text{Fe}^{3+}/\text{Mn}^{2+}/\text{CH}_3\text{COO}^-$
3. FLAME TEST Paste of the given sample in conc. HCl was ignited in bunsen flame.	Bluish green.	Cu^{2+}
	Crimson red (persistent).	Sr^{2+}
	Brick red (non-persistent).	Ca^{2+}
	Bluish white flash.	Pb^{2+}
	Grassy green.	Ba^{2+}
	No specific colour.	$\text{Cu}^{2+}/\text{Sr}^{2+}/\text{Ca}^{2+}/\text{Pb}^{2+}/\text{Ba}^{2+}$ absent.
4. Dil. H_2SO_4 TEST A pinch of sample + dil. H_2SO_4 ; warm the contents.	Brisk effervescence. Colourless, odourless gas turns lime water milky.	CO_3^{2-} confirmed.
	Colourless, burning smelling gas turns acidified $\text{K}_2\text{Cr}_2\text{O}_7$ paper/solution green.	SO_3^{2-} present.
	Colourless gas with rotten egg smell turns lead-acetate paper black.	S^{2-} present.
	No gas evolved.	$\text{CO}_3^{2-}, \text{SO}_3^{2-}, \text{S}^{2-}$ are absent.
5. Conc. H_2SO_4 TEST A pinch of sample + conc. H_2SO_4 , warm the contents, if necessary.	Colourless pungent smelling gas gives white fumes when a glass rod dipped in NH_4OH is brought near it.	Cl^- present.
	Brown gas when Cu turnings/paper pallet is added.	NO_3^- present.
	Colourless, vinegar smelling gas turns blue.	CH_3COO^- present.
	No gas evolved.	$\text{Cl}^-, \text{NO}_3^-, \text{CH}_3\text{COO}^-$ absent.
6. TEST FOR PO_4^{3-} Sample+conc. HNO_3 , boil and add ammonium molybdate solution excess. Allow to stand.	A canary yellow precipitate.	PO_4^{3-} confirmed.
7. TEST FOR SO_4^{2-} Sample + dil. HCl. Boil and add BaCl_2 solution.	White precipitate insoluble in HCl.	SO_4^{2-} confirmed.
8. CONFIRMARY TEST FOR Cl^- ION a) Sample solution + dil. $\text{HNO}_3 + \text{AgNO}_3$ solution.	Curdy white precipitate soluble in excess NH_4OH .	Cl^- confirmed.
b) Sample(solid) + $\text{K}_2\text{Cr}_2\text{O}_7$ (1:1) + 2ml conc. H_2SO_4 . Heat gently Pass the red vapour through NaOH solution.	Red vapour is formed. Yellow colouration of the solution.	Cl^- confirmed. Cl^- confirmed.
Add Pb-acetate solution to the yellow coloured solution.	Yellow precipitate.	Cl^- confirmed.
c) Sample solution + sodium nitroprusside solution.	Violet/purple colouration.	S^{2-} confirmed.
d) Sample solution + freshly prepared FeSO_4 solution (1:1) + conc. H_2SO_4 along the wall of the test tube.	Brown ring in the middle.	NO_3^- confirmed.
e) Water extract (shake a little of the sample with water) + neutral FeCl_3 solution; boil and allow to stand.	Reddish brown precipitate.	CH_3COO^- confirmed.

9. Group separation: Prepare original solution (~40ml) of the sample/salt in water (boil/ add dil. HCl if required) in the boiling tube.		
a) OS (little amount) + Nessler's reagent.	Brown precipitate.	Gr. 0 (NH_4^+) confirmed.
b) OS + dil. HCl	White precipitate.	Gr. I present (Pb^{2+} present).
c) OS + dil. HCl + H_2S	Black/yellow precipitate.	Gr. II present ($\text{Cu}^{2+}/\text{Cd}^{2+}$).
* d) OS + NH_4Cl (solid) + NH_4OH excess; till smells ammonia, mix well.	Gelatinous white/brown precipitate.	Gr. III present ($\text{Al}^{3+}/\text{Fe}^{3+}$).
e) OS + NH_4OH + H_2S	Black/flesh coloured/white (dirty) precipitate.	Gr. IV present ($\text{Ni}^{2+}/\text{Mn}^{2+}/\text{Zn}^{2+}$).
f) OS + NH_4OH + saturated $(\text{NH}_4)_2\text{CO}_3$ solution.	White precipitate.	Gr. V present ($\text{Ba}^{2+}/\text{Sr}^{2+}/\text{Ca}^{2+}$).
g) OS + NH_4OH + $(\text{NH}_4)_3\text{PO}_4$. Shake and scratch the inner wall of the test tube with glass rod.	White crystalline precipitate.	Gr. VI present (Mg^{2+}) confirmed.
10. Confirmatory tests for Cations		
For NH_4^+		
a) Sample + NaOH solution and warm.	Ammoniacal smelling gas gives white fumes when a rod dipped in HCl is brought.	NH_4^+ confirmed.
b) SS + Nessler's reagent.	Brown precipitate.	NH_4^+ confirmed.
For Pb^{2+} : Dissolve Gr. I precipitate in boiling water.		
a) Precipitate solution + KI solution.	Golden-yellow precipitate.	Pb^{2+} confirmed.
b) Precipitate solution + H_2SO_4 .	White precipitate.	Pb^{2+} confirmed.
For $\text{Cu}^{2+}/\text{Cd}^{2+}$: Dissolve Gr. II precipitate (black/yellow) in 50% HNO_3 by boiling.		
a) Precipitate solution + NH_4OH excess.	A deep blue colouration.	Cu^{2+} confirmed.
b) Precipitate solution + $\text{K}_4[\text{Fe}(\text{CN})_6]$ solution.	a) Chocolate brown precipitate.	Cu^{2+} confirmed.
	b) Bluish-white precipitate.	Cd^{2+} confirmed.
For $\text{Al}^{3+}/\text{Fe}^{3+}$: Dissolve Gr. III precipitate in dil. HCl by warming.		
a) Precipitate solution + few drops of blue litmus solution + NH_4OH excess.	A blue precipitate floating in the colourless solution.	Al^{3+} confirmed.
b) Precipitate solution + $\text{K}_4[\text{Fe}(\text{CN})_6]$.	Persian blue colouration.	Fe^{3+} confirmed.
c) Precipitate solution + NH_4SCN solution.	Blood red colouration.	Fe^{3+} confirmed.
For $\text{Ni}^{2+}/\text{Zn}^{2+}$: Dissolve black/white precipitate of Gr. IV in aqua regia by heating and add water.		
a) Precipitate soln + 3 drops DMG solution + excess NH_4OH .	Rose red precipitate/colouration.	Ni^{2+} confirmed.
b) Precipitate solution + $\text{K}_4[\text{Fe}(\text{CN})_6]$.	White/bluish white precipitate.	Zn^{2+} confirmed.
For Mn^{2+}		
Flesh coloured Gr. IV precipitate + conc. HNO_3 excess + PbO_2 ; heat the contents.	Purple colouration of the supernatant liquid.	Mn^{2+} confirmed.
For $\text{Ba}^{2+}/\text{Sr}^{2+}/\text{Ca}^{3+}$: Dissolve Gr. V precipitate in minimum amount of dil. CH_3COOH by boiling till effervescence ceases.		
a) Precipitate solution + K_2CrO_4 solution.	Yellow precipitate.	Ba^{2+} confirmed.
b) Precipitate solution + $(\text{NH}_4)_2\text{SO}_4$ solution.	White precipitate.	Sr^{2+} confirmed.
c) Precipitate solution + $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution.	Crystalline white precipitate.	Ca^{3+} confirmed.

* In group III, if the salt is light green, precipitate will be formed by adding the HNO_3 in the sample and heating it then mixing with NH_4Cl and NH_4OH excess.

RESULT: