Salt Analysis

(20) $HCl + NaOH \rightarrow Na^+Cl^- + H_2O$

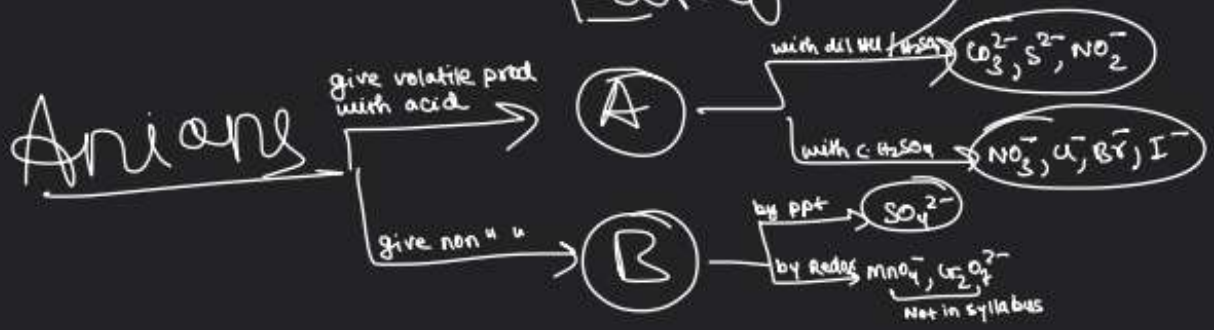
- Na^+ → Basic radical (from base) [Cation]
- Cl^- → Acid radical (from acid) [Anion]

(21) Anions are always tested before cations → because some anions interfere with the tests of cations.

Interfering anions

$C_2O_4^{2-}$, PO_4^{3-}, AsO_4^{3-}, SiO_3^{2-}, F^-, BO_3^{2-}

(Fir Bola Ox Aise Sekho Fatafat)



Preliminary test:

CO_3^{2-}, NO_2^-, S^{2-}

dry salt (S) $\xrightarrow{\text{dil. HCl or dil } H_2SO_4}$

- colourless, odourless, with effervescence → $CO_2(g)$ → may be CO_3^{2-}
  - → turns lime water milky: $CO_2 + Ca(OH)_2 \rightarrow CaCO_3\downarrow$
- Rotten egg smell → $H_2S(g)$ → S^{2-}
  - → turns lead acetate paper black
  - $H_2S + Pb(CH_3COO)_2 \rightarrow PbS\downarrow$ (black)
- Brown colour → $NO_2(g)$ → NO_2^-
  - → turns starch acidified KI solution blue
  - $NO_2 + KI + \text{starch} + CH_3COOH \rightarrow$ Blue

NO_3^-, Cl^-, Br^-, I^-

dry salt $\xrightarrow{\text{Conc. } H_2SO_4}$

- Colourless pungent smell gas → gives white fumes with NH_4OH
  - (gas → HCl ↑, possible anion → Cl^-)
  - $NH_4OH + HCl \rightarrow NH_4Cl$ (white fumes)
- Reddish brown gas
  - Intensity of gas ↑ on addition with MnO_4
  - (gas → Br_2, possible anion → Br^-)
- Violet gas →
  - Intensity of gas ↑ with MnO_2
  - (gas → I_2, possible anion → I^-)
- Brown gas →
  - Intensity of gas ↑ with addition of Cu turning → Blue soln
  - (gas → NO_2, possible anion → NO_3^-)

diff b/w brown gas of Br_2 and NO_2

$Br_2 \xrightarrow{H_2O}$ brown soln

$NO_2 \xrightarrow{H_2O}$ colourless

$(NO + HNO_3$, $+2$, $+5)$

↓

Disproportionation

Confirmatory Tests

dry salt → if soluble then use water extract salt

dry salt → if insoluble then make it soluble by adding Na_2CO_3

$M^+X^- + Na_2CO_3 \rightarrow Na^+X^- + M_2CO_3$
(w/i) (w/s) (sodium extract)

CO_3^{2-}

① $CO_3^{2\ominus}$ (dry salt) $+ dil.\ H_2SO_4 \rightarrow CO_2\uparrow$

- lime water $Ca(OH)_2 \rightarrow CaCO_3\downarrow$ (turns milky)
- $Ba(OH)_2 \rightarrow BaCO_3\downarrow$ (W) — Baryta water

② $CO_3^{2\ominus}$ (w/s) $+ BaCl_2 / CaCl_2$ (w/s) → $BaCO_3 / CaCO_3$ (w/i) white ppt

- $BaCO_3$ + HCl → $BaCl_2 / CaCl_2$ (w/s)
- $BaCO_3$ + HNO_3 → $BaNO_3 / CaNO_3$ (w/s)

③ $CO_3^{2\ominus} + AgNO_3 \rightarrow Ag_2CO_3$

- Ag_2CO_3 —Δ→ $Ag_2O\downarrow$ (brown ppt) —Δ→ $2Ag\downarrow$ (black ppt)
- Ag_2CO_3 + HNO_3 → $AgNO_3$ w/s
- Ag_2CO_3 + HCl → $AgCl\downarrow$ white ppt
- Ag_2CO_3 + excess NH_3 → $[Ag(NH_3)_2]^+$ complex

S^{2-}

① S^{2-} (Salt) + dil HCl / dil H_2SO_4 → $H_2S\uparrow$
- → lead acetate → PbS↓ (black ppt)
- → cadmium acetate → CdS↓ (yellow ppt)

② S^{2-} (soluble Salt) (Na_2S) → + $Na_2[Fe(CN)_5NO]$ → $Na_4[Fe(CN)_5NOS]$ (violet↓)

③ ppt test

S^{2-} (Soluble)
- $AgNO_3$ → Ag_2S↓
- $Pb(NO_3)_2$ → PbS↓ } black
- $Pb(CH_3COO)_2$ → PbS↓ } black
- Cd^{+2} → CdS↓ } yellow
- $Hg(NO_3)_2$ → HgS↓ } black

① Redox test

S^{2-} (R.A)
- IO_3^- (colourless) → I_2 (violet)
- $Cr_2O_7^{2-}$ (orange) → Cr^{3+} (green)
- MnO_4^- (violet) → Mn^{2+} (colourless)
- Fe^{3+} (yellow) → Fe^{2+} (green)
- Cl_2 water (green yellow) → Cl^- (colourless)
- Br_2 water (Br_2 brown) → Br^- (colourless)

NO_2^-

① Salt (NO_2^-) + dil H_2SO_4 $\xrightarrow{\Delta}$ $NO_2\uparrow$

NO_2 → KI + starch + CH_3COOH → Blue

Reason of blue colour

$NO_2^- + CH_3COOH \rightarrow HNO_2$

HNO_2 (CA) $+ KI^- \rightarrow I_2^0 + NO$

I_2 + starch → blue

② Griess Ilosvay test

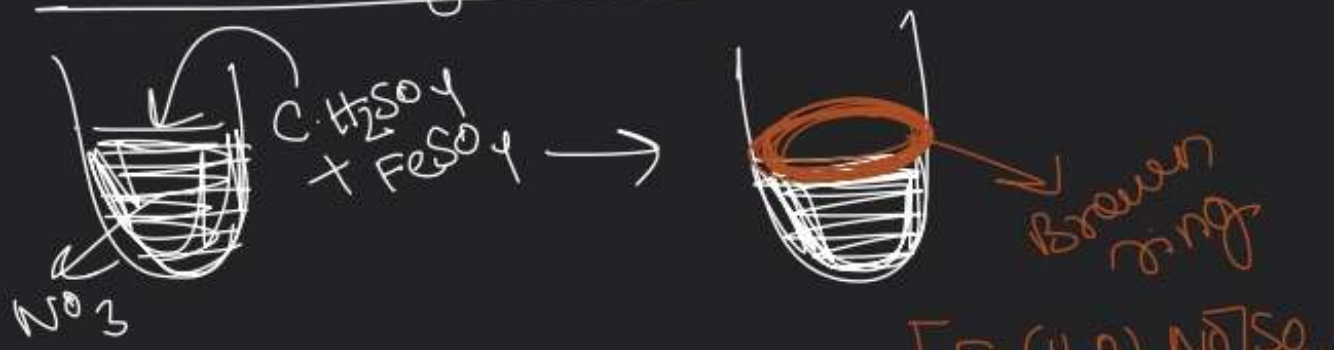
NO_2^- (W/S salt) → Sulphanilic acid + Naphthylamine + Acetic acid → Red azo

$NO_3^{\ominus}$

① $NO_3^{\ominus} \xrightarrow{c.H_2SO_4}$ $NO_2\uparrow$
(salt) (brown gas)

$Cu_{turning} + NO_3^- + c.H_2SO_4 \rightarrow \underset{(blue)}{CuSO_4} + NO + O_2$
$NO_2\uparrow$

② Brown ring test



$[Fe(H_2O)_5NO]SO_4$

Rxns

$NO_3^{\ominus} + c.H_2SO_4 \rightarrow HNO_3$

(Key step) $HNO_3 + FeSO_4 \rightarrow Fe^{3+} + NO$

$FeSO_4(aq) + NO \rightarrow$

Common tests for Br^-, Cl^-, I^-

with $AgNO_3$

$Cl^- + AgNO_3 \rightarrow AgCl\downarrow$ (white ppt)

- CN^- → $[Ag(CN)_2]^-$ sol.
- $S_2O_3^{2-}$ → $[Ag(S_2O_3)_2]^{3-}$ sol.
- Excess NH_3 → $[Ag(NH_3)_2]^+$ sol.

$Br^- + AgNO_3 \rightarrow AgBr\downarrow$ (pale yellow)

- → ↓
- → ↓
- Excess NH_3 → partial soluble

$I^- + AgNO_3 \rightarrow AgI\downarrow$ (yellow)

- → ↓
- → ↓
- → insoluble

with $Pb(NO_3)_2$

$Cl^- + Pb(NO_3)_2 \rightarrow PbCl_2\downarrow$ (W) (sol.^{y} in warm water)

$Br^- + Pb(NO_3)_2 \rightarrow PbBr_2\downarrow$ (Y) (sol.^{y} in hot water)

$I^- + Pb(NO_3)_2 \rightarrow PbI_2\downarrow$ (Y) (sol.^{y} in boiling water)

Special test for Cl^-

Chromyl chloride test

$$Cl^- + Cr_2O_7^{2-} \xrightarrow{+ conc.\ H_2SO_4} CrO_2Cl_2\uparrow$$
(brown)

CrO_2Cl_2 $\xrightarrow{NaOH}$ Na_2CrO_4 (yellow soln)

for removal of extra NaOH

Na$_2$CrO$_4$ → part (A) and part (B)

part (A) → add $(CH_3COO)_2Pb$ → $PbCrO_4$ (yellow ppt) chrome yellow

part (B) → add H_2O + dil. H_2SO_4 → CrO_5

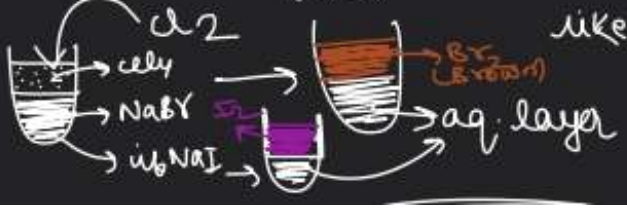
Special test for Br^-/I^-

(layer test)

$Br^- + Cl_2 \rightarrow Br_2$ (brown) $+ Cl^-$

$I^- + Cl_2 \rightarrow I_2$ (violet) $+ Cl^-$

Br_2/I_2 soluble in non polar solvents like CCl_4.



SO_4^{2-}

SO_4^{2-} → $BaCl_2$ → $BaSO_4\downarrow$ (insol in HCl and HNO_3)

SO_4^{2-} → $+(CH_3COO)_2Pb$ → $PbSO_4\downarrow$ (white ppt)

↓

Soluble by Sodium extract

Cations

when cold	when hot	ion
white	yellow	Zn^{2+}
blue	white	Cu^{2+}
pink	blue	Co^{2+}

Fe^{3+} → Brown

Ni^{2+} → Bright green

1) Flame test

For s-block elements

Be Mg

XX no flame test (due to high I.E)

Ca → Brick red

Ba → Apple green

Li → Crimson red

Na → golden yellow

Cu^{2+} → green flame with blue centre

2) Borax Bead Test (For transition metals)

$Na_2B_4O_7 \cdot 10H_2O$

$\xrightarrow{\Delta} Na_2B_4O_7$

$\downarrow \Delta$

$NaBO_2 + B_2O_3$

(Sodium metaborate) (Basic anhydride)

Transparent glassy bead

↓ Sample salt

Coloured metaborate

Eg:- $Cr(BO_2)_3$ → green

$Co(BO_2)_2$ → blue

Borax $\xrightarrow{\Delta}$ Sample salt → coloured bead

Oxidising flame (non luminous)

Reducing flame (luminous)

Ox		red		
cold	hot	cold	hot	
blue	green	red	colourless	Cu^{2+}
Brown	Violet	grey	grey	Ni^{2+}
yellow	brown	green	green	Fe^{3+}

3) Phosphate bead test (Microcosmic salt)

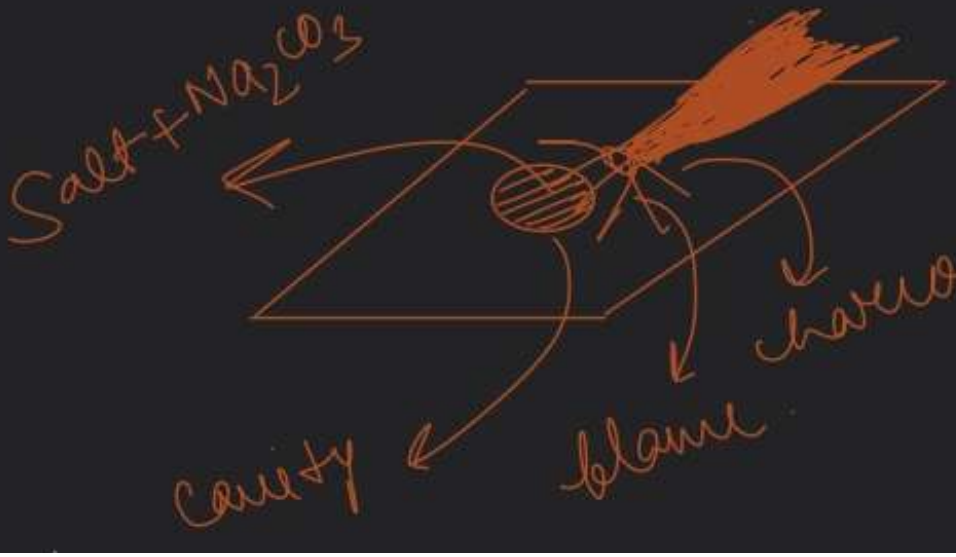
H_3PO_4 $\xrightarrow{-2H^+}$ HPO_4^- (Hydrogen phosphate ion)

$HPO_4^- \xrightarrow{+NH_4^+ \uparrow,\ +Na^+}$ $NaNH_4HPO_4 \cdot 4H_2O$

$NaNH_4HPO_4 \cdot 4H_2O \xrightarrow{\Delta}$ $NaPO_3$ + $NH_3\uparrow$ + H_2O

$NaPO_3$ → Sodium meta phosphate → Bead → same as Borax bead

4) Charcoal Cavity test



Salt + Na_2CO_3 + Δ → metal oxide (Give colour)

hot	cold	
Yellow	grey	Pb^{2+}
Yellow	white	Zn^{2+}

5) **Cobalt nitrate charcoal cavity test.**

if white residue is obtained in pber test then add 2-3 drops of $Co(NO_3)_2$

White residue + $Co(NO_3)_2 \xrightarrow{\Delta}$ CoO·MO (MO ← metal)

$\downarrow$

$CoO + (NO_2 + O_2)\uparrow$

- CoO·ZnO → Green
- CoO·Al_2O_3 → Blue
- CoO·MgO → Pink

Classification of cation

Gr – I ↓ Gr – 0: Ksp ↑, Solubility ↑, Ionization ↑

Group	Reagent	Cations
Gr I	(dil HCl)	Ag^+ (Aaj), Hg_2^{2+} (Hoga), Pb^{2+} (Prabhat)
Gr II → IIA	(H_2S in P.O dil HCl)	Hg^{2+} (Hogi), Pb^{2+} (Punjabi), Cu^{2+} (cudiyan), Cd^{2+} (Seedi), Bi^{3+} (Bindass)
Gr II → IIB		As^{3+}/As^{5+} (Aisa), Sb^{3+}/Sb^{5+} (Sala), Sn^{2+}/Sn^{4+} TanaTan
Gr III	(NH_4OH in P.O NH_4Cl)	Al^{3+} (Alu), Fe^{3+} (Feka), Cr^{3+} (Caro)
Gr IV	(H_2S in P.O NH_4OH)	Ni^{2+} (Ni), Co^{2+} (Co), Mn^{2+} Zn^{2+} (mayan)
Gr V	($(NH_4)_2CO_3$ in P.O NH_4Cl)	Ba^{2+} (Bharat), Sr^{2+} (Sar), Ca^{2+} (Car)
Gr VI	(No specific reagent)	Na^+ (Na), Mg^{2+} (mango), K^+ (Kar)
Gr 0		NH_4^+

Confirmatory test for grup-I

Salt + dil HCl → white ppt ($AgCl$ / Hg_2Cl_2 / $PbCl_2$)

↓ A

Soluble
Pb^{2+} present
($PbCl_2$ is soluble)

Insoluble
(Hg_2^{2+} / Ag^+)

↓ Excess NH_4OH

Insoluble (Hg_2^{2+}) → Black ppt for a long time

Soluble $[Ag(NH_3)_2]^+$

Test for Pb^{2+}

$PbCl_2$

- * KI → PbI_2 ↓ (yellow)
- * K_2CrO_4 → $PbCrO_4$ ↓ (yellow) —NaOH Excess→ $Na_2[Pb(OH)_4]$
- H_2S → PbS ↓ (black)
- dil H_2SO_4 → $PbSO_4$ ↓ (white) — NH_4OH (dil) → $(NH_4)_2[PbO_2]$ + (SO_4^{2-})
- KCN → $Pb(CN)_2$ ↓ (white)

Test for grp II

(II B) ↑ sol; (II A) ↑ insol (CuS, PbS)

Salt + H_2S (in PO dil HCl) ⟶ ppt $\xrightarrow{(NH_4)_2S}$

Test for Pb^{2+}

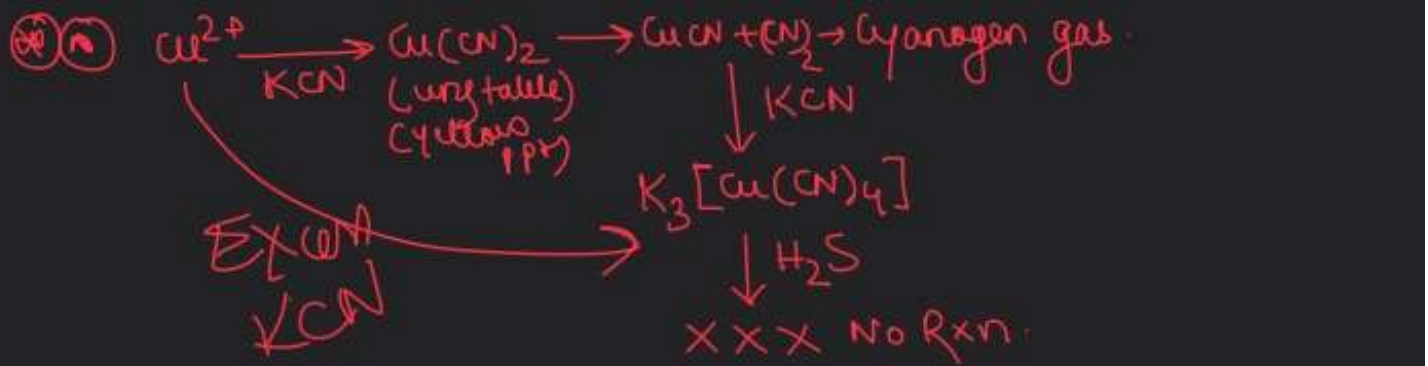
PbS (black ppt) $\xrightarrow{\text{dil } HNO_3}$ $Pb(NO_3)_2$ (sol^n) $\xrightarrow{\text{dil } H_2SO_4}$ $PbSO_4$ (white ppt)

$PbSO_4$ $\xrightarrow{NH_4(CH_3COO)}$ $(NH_4)_2[Pb(CH_3COO)_4]$ (sol) $\xrightarrow{K_2CrO_4}$ $PbCrO_4$↓ (yellow ppt) $\xrightarrow{NaOH}$ $Na_2[Pb(OH)_4]$ sol

Test for Cu^{2+}

CuS black ppt $\xrightarrow{\text{dil } HNO_3}$ $Cu(NO_3)_2$ Soluble $\xrightarrow[\text{long time}]{\Delta}$ $CuSO_4$ (solution)

$CuSO_4$ $\xrightarrow{NH_4OH}$ $Cu(OH)_2 \cdot CuSO_4$↓ (Basic copper sulphate) $\xrightarrow{\text{Excess } NH_4OH / \text{ or } NH_3 \text{ soln}}$ $[Cu(NH_3)_4]SO_4$ $\xrightarrow{CH_3COOH}$ $CuSO_4$ (bluey soln) $\xrightarrow{K_4[Fe(CN)_6]}$ ↓ $Cu_2[Fe(CN)_6]$ (chocolate brown ppt)



Test for Grp III

Salt + NH4OH → ppt

↑

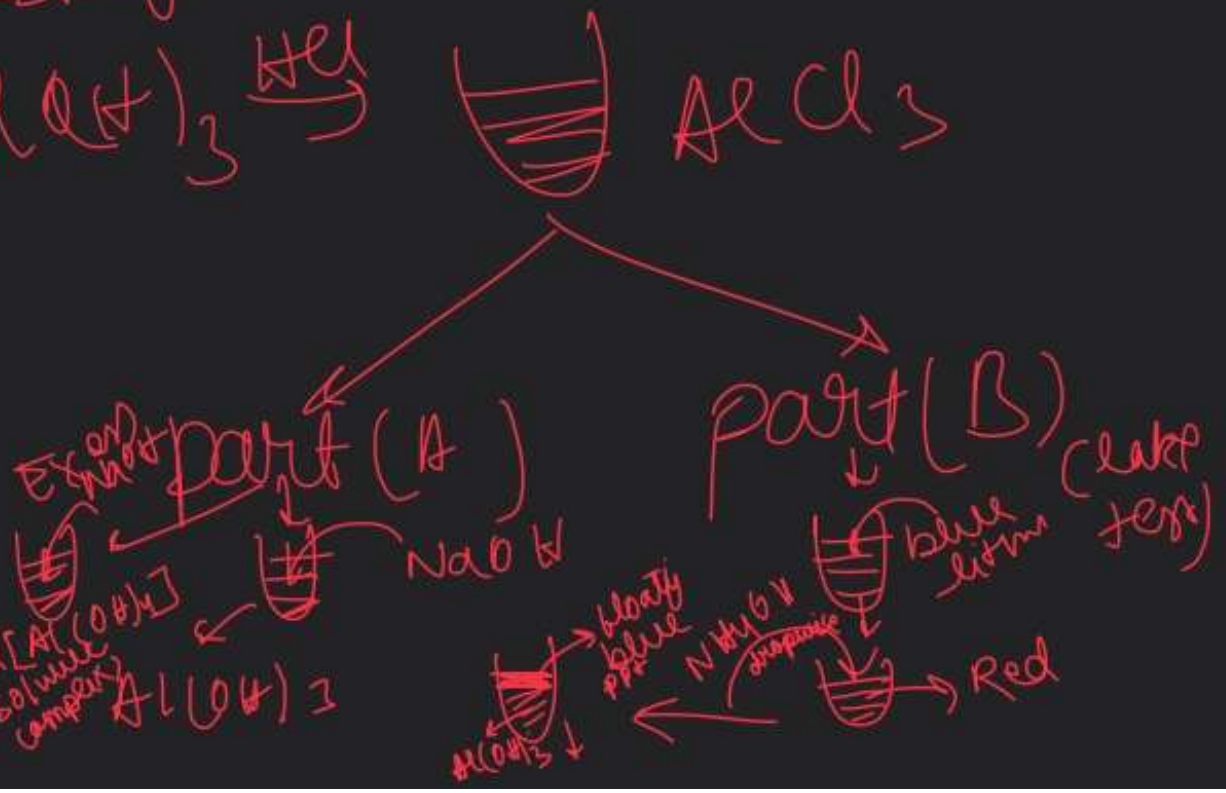
NH4Cl

$Al(OH)_3$ → white gel ppt

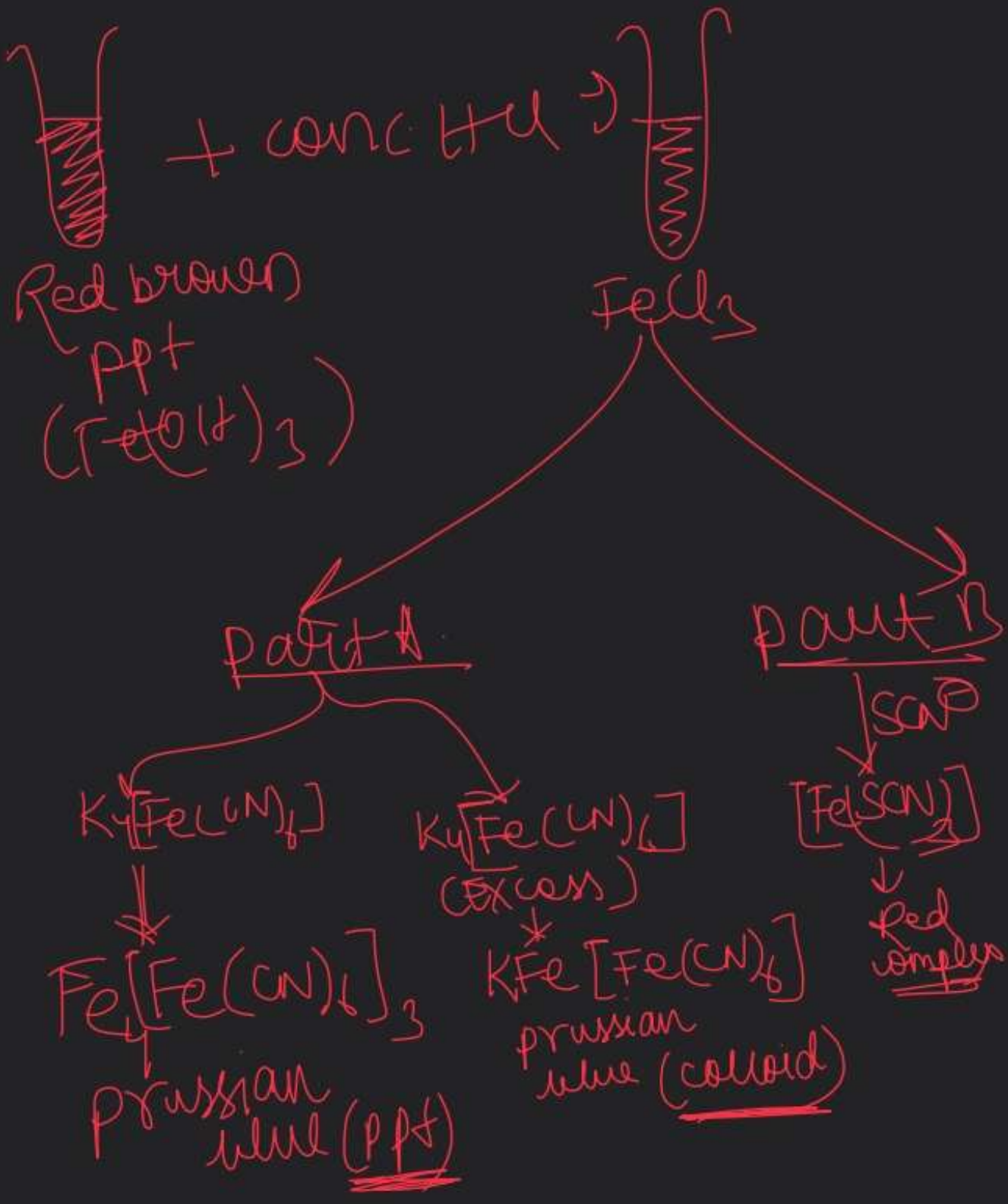
$Fe(OH)_3$ → Red brown ppt

~~Test for Al^{3+}~~

$Al(OH)_3$ $\xrightarrow{HCl}$ $AlCl_3$



Test for Fe^{3+}


+ conc HCl
Red brown ppt ($Fe(OH)_3$)
$FeCl_3$
part A
part B
$K_4[Fe(CN)_6]$
$K_4[Fe(CN)_6]$ (excess)
SCN
$[Fe(SCN)]$
red complex
$Fe_4[Fe(CN)_6]_3$
prussian blue (PPt)
$KFe[Fe(CN)_6]$
prussian blue (colloid)

Test for Group IV

OS + NH_4OH + H_2S → ppt

NiS, MnS, ZnS, CoS

- Soluble: Mn^{2+}, Zn^{2+}
- insoluble: (NiS, CoS)
  - aqua regia
    - green: Ni^{2+}
    - pink: Co^{2+}

Test for Zn^{2+}

ZnS ↓ —dil HCl / Δ→ $ZnCl_2$ (Sol/mole)

- Part (a)
  - NaOH → $Zn(OH)_2$
  - Excess NaOH → $Na_2[Zn(OH)_4]$
- Part (b) → $K_4[Fe(CN)_6]$ → $Zn_2[Fe(CN)_6]$ white ppt

Test for Ni^{2+}

NiS —Aqua Regia→ $NiCl_2$ (Sol) —NH_4OH dmy→ Rosy red ppt ↓ or unstyred ppt ↓

Test from Grp V

O.S + $(NH_4)_2CO_3$ + NH_4Cl → white ppt ($BaCO_3$ / $SrCO_3$ / $CaCO_3$)

$\xrightarrow{CH_3COOH}$ all sol

↓

divide into 3 parts

Part (a) $\xrightarrow{K_2CrO_4}$ $BaCrO_4 \downarrow$ (yellow) → Ba^{2+} ✓

Part (b) $\xrightarrow{(NH_4)_2SO_4}$ (white) $SrSO_4 \downarrow$ → Sr^{2+} ✓

Part (c) $\xrightarrow{(NH_4)_2C_2O_4}$ $CaC_2O_4 \downarrow$ (white) → Ca^{2+} ✓

Ba — CrO_4^{2-}, SO_4^{2-}, $C_2O_4^{2-}$ — Insol

Sr — CrO_4^{2-} → sol; SO_4^{2-}, $C_2O_4^{2-}$ — insol

Ca — $C_2O_4^{2-}$ → insol; SO_4^{2-}, CrO_4^{2-} → sol

Grp VI

test of Mg^{2+}

Filtrate of grp V $\xrightarrow{Na_2HPO_4}$ $MgNH_4PO_4 \downarrow$ (white ppt)

NH_4^+ Grp 0

→ do not use prev filtrate

→ Always use O.S