

University of Tikrit College Of Pharmacy



# **General Inorganic and Analytical Chemistry**

First Year Students Practical Part 1 And 2

By Prof. Subhi A. Al-Jibori And Dr. Sermed B. Dikram



# Demonstration of Some Laboratory Equipments.



Group Separation and Identification of the Cations

Group I Cations (The Silver Group) Ag<sup>+</sup>, pb<sup>2+</sup> and Hg<sub>2</sub><sup>2+</sup> ions

Reactions of the lead ion, pb<sup>2+</sup>:

1. With dilute HCl

White precipitate of pbCl<sub>2</sub> is formed.

 $pb (NO_3)_2 + 2HCl \longrightarrow pbCl_2 \downarrow + 2HNO_3$ 

(white)

or  $pb^{2+} + 2Cl^- \longrightarrow pbCl_2 \bigvee$ 

 $pbCl_2$  is soluble in hot water, but separates out again in needles when the solution is cooled.

2. With KI solution

0

Yellow precipitate of pbI<sub>2</sub> is formed.

 $pb(NO_3)_2 + 2KI \longrightarrow pbI_2 \downarrow + 2KNO_3$ (yellow)

 $pbI_2$  is soluble in excess of KI solution forming a complex ion.  $pbI_2 + 2KI \iff K_2 [pbI_4]$ 

#### 3. With dilute $H_2SO_4$

White precipitate of pbSO<sub>4</sub> is formed.

 $pb (NO_3)_2 + H_2SO_4 \longrightarrow pbSO_4 \checkmark + 2HNO_3$ (white)

 $pbSO_4$  is soluble in a concentrated solution of ammonium acetate.  $pbSO_4 + 2NH_4.C_2H_3O_2 \longrightarrow pb(C_2H_3O_2)_2 + (NH_4)_2 SO_4$ soluble

#### 4. With $K_2CrO_4$ solution

Yellow precipitate of pbCrO<sub>4</sub> is formed.

 $pb (NO_3)_2 + K_2CrO_4 \longrightarrow pbCrO_4 + 2KNO_3$ (yellow)

 $pbCrO_4$  is soluble in acetic acid and in ammonia solution, but soluble in alkali hydroxides and in nitric acid.

 $pbCrO_4 + 4NaOH \longrightarrow Na_2 [pbO_2] + Na_2CrO_4 + 2H_2O$ sodium plumbite

 $pbCrO_4 + H_3O^+ \longrightarrow pb^{2+} + HCrO_4 + 2H_2O$ 

5. With sulphide ion solution (use H<sub>2</sub>S)

Black precipitate of pbS is formed.

 $pb (NO_3)_2 + H_2S \longrightarrow pbS \downarrow + 2HNO_3$ (black)

pbS is soluble in hot dilute  $HNO_3$ .

 $3pbS + 8HNO_3 \longrightarrow 3pb(NO_3)_2 + 2NO + 4H_2O + 3S$ <u>Note</u>:  $H_2S$  is evolved from thioacidamide. Which is hydrolyzed rapidly in acidified aqueous solution (dil HCl) and librate  $H_2S$  after heating the mixture in a water bath.

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$$CH_3 - C - NH_2 + H_2O \longrightarrow CH_3 - C - NH_2 + H_2S^{\uparrow}$$

#### 6. With NaOH solution

White precipitate of  $pb(OH)_2$  is formed.

 $pb (NO_3)_2 + 2NaOH \longrightarrow pb(OH)_2 \checkmark + 2NaNO_3$ (white)

 $pb(OH)_2$  is soluble in excess of the reagent.

 $pb(OH)_2 + 2NaOH \longrightarrow Na_2 [pbO_2] + 2H_2O$ 

<u>Reactions of the mercurous ion,  $Hg_2^{2+}$ :</u>

1. With dilute HCl

White precipitate of Hg<sub>2</sub>Cl<sub>2</sub> (Calomel) is formed.

 $Hg_{2}(NO_{3})_{2} + 2HC1 \longrightarrow Hg_{2}Cl_{2} \checkmark + 2HNO_{3}$ (white)

 $Hg_2Cl_2$  is insoluble in hot water, but soluble in aqua regia (1ml of concentration  $HNO_3 + 3ml$  of concentration HCl).

 $HNO_{3} + 3HC1 \longrightarrow NOC1 + 2C1 + 2H_{2}O$  $Hg_{2}Cl_{2} + 2C1 \longrightarrow 2HgCl_{2}$ soluble

Hg<sub>2</sub>Cl<sub>2</sub> turns to a black mixture when treated with aqueous ammonia.

 $Hg_{2}Cl_{2} + 2NH_{3} \longrightarrow Hg(NH_{2})Cl \checkmark + Hg \checkmark + NH_{4}Cl$ (white) (black)

#### 2. With KI solution

Yellowish-green precipitate of  $Hg_2I_2$  is formed.  $Hg_2(NO_3)_2 + 2KI \longrightarrow Hg_2I_2 \checkmark + 2KNO_3$ (yellowish-green)

 $Hg_2I_2$  is soluble in excess of KI solution.

 $Hg_2I_2 + 2KI \longleftarrow K_2 [HgI_4] + Hg$ 

Potassium mercuri-iodide

#### 3. With $K_2CrO_4$ solution

Brown a morphous precipitate of  $Hg_2CrO_4$  is formed, which is converted into a red crystalline form on boiling.

 $Hg_{2} (NO_{3})_{2} + K_{2}CrO_{4} + 2KNO_{3}$ (brown)

#### 4. With NaOH solution

Black precipitate of Hg<sub>2</sub>O is formed.

 $Hg_2(NO_3)_2 + 2NaOH \longrightarrow Hg_2O \downarrow + 2NaNO_3 + H_2O$ (black)

#### 5. With ammonia solution

Finely-divided black precipitate of Hg is formed.

$$2Hg_2(NO_3)_2 + 4NH_3 + H_2O \longrightarrow O NH_2.NO_3 + 2Hg + 2NH_4NO_3$$
  
Hg (black)

#### 6. With $SnCl_2$ solution

Finely-divided black precipitate of Hg is formed with excess of the reagent.

 $Hg_{2}(NO_{3})_{2} + SnCl_{2} + 2HCl \longrightarrow 2Hg \checkmark + SnCl_{4} + 2HNO_{3}$ (black)

#### Reactions of the Silver ion, Ag<sup>+</sup>:

1. With dilute HCl

White precipitate of AgCl is formed.

 $AgNO_3 + HCl \longrightarrow AgCl + HNO_3$ (white)

AgCl is insoluble in dilute HNO<sub>3</sub>, but is soluble in aqueous ammonia owing to the formation of the silver diammino complex

ion, Ag(NH<sub>3</sub>)<sub>2</sub><sup>+</sup>

 $AgCl + 2NH_3 \longrightarrow Ag(NH_3)_2Cl$ 

AgCl is precipitated again from the ammonical solution by the addition of dilute HNO<sub>3</sub>

 $Ag(NH_3)_2Cl + 2HNO_3$  AgCl  $\downarrow + 2NH_4NO_3$ 

2. With KI solution

Yellow precipitate of AgI is formed.

$$AgNO_3 + KI \longrightarrow AgI + KNO_3$$
  
(yellow)

AgI is insoluble in concentrated ammonia solution, but is soluble in solutions of KCN or  $Na_2S_2O_3$ 

AgI + KCN
$$\leftarrow$$
 K[Ag (CN)2] + KIAgI + 2 Na2S2O3 $\leftarrow$  Na3[Ag (S2O3)2] + NaISodium thiosulphate $\sim$  Sodium thiosulphate

3. With  $K_2CrO_4$  solution

Red precipitate of  $Ag_2CrO_4$  is formed.

 $2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 \downarrow + 2KNO_3$ (red)

4. With NaOH solution

Brown precipitate of Ag<sub>2</sub>O is formed.

 $2AgNO_3 + NaOH \longrightarrow Ag_2OV + 2NaNO_3 + H_2O$ (brown)

#### 5. With ammonia solution

White precipitate at first, which quickly passes into brown  $Ag_2O$   $Ag_2O$  is soluble in excess of the reagent.

 $Ag_2O + 4NH_3 + H_2O \longrightarrow 2[Ag(NH_3)_2]OH$ 



# Analysis of the silver group (group 1)



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# Separation and identification of Group I cations

To the given solution in a test tube add dilute HCl in excess and filter. Wash the ppt., which may contain pbCl<sub>2</sub>, AgCl and Hg<sub>2</sub>Cl<sub>2</sub> with a little very dilute HCl.



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#### **Group II cations**

 The copper group (group II A) Hg<sup>2+</sup>, Bi<sup>3+</sup>, Cu<sup>2+</sup> and Cd<sup>2+</sup> ions.
 The Arsenic group (group II B)

#### The Copper Group II A

# Reactions of the mercuric ion, Hg<sup>2+</sup>

1. With sulphide ion (use  $H_2S$ )

Black precipitate of HgS is formed.

 $HgCl_{2} + 2H_{2}S \longrightarrow Hg_{3}S_{2}Cl_{2} + 4HCl$  $Hg_{3}S_{2}Cl_{2} + H_{2}S \longrightarrow 2HCl + 3HgS \checkmark$ 

(black)

the net result is HgCl<sub>2</sub> + H<sub>2</sub>S  $\longrightarrow$  HgS $\downarrow$ + 2HCl

HgS is soluble in aqua regia.

 $3HgS + 2HNO_3 + 6HCl \longrightarrow 3HgCl_2 + 2NO + 3S + 4H_2O$ 

#### 2. With SnCl<sub>2</sub> solution

White precipitate of HgCl<sub>2</sub> is formed.

 $2\text{HgCl}_2 + \text{SnCl}_2 \longrightarrow \text{SnCl}_4 + \text{Hg}_2\text{Cl}_2 \bigvee$ (white)

 $Hg_2Cl_2$  is reduced by the excess of the reagent to grey-black metallic mercury (after 5 minutes).

 $Hg_2Cl_2 + SnCl_2 \implies SnCl_4 + 2Hg \downarrow$ (black)

#### 3. With NaOH solution

Reddish-brown precipitate is formed initially converted to yellow precipitate of HgO.

 $HgCl_2 + 2NaOH \longrightarrow HgO + 2NaCl + H_2O$ (yellow)

### 4. With ammonia solution

White precipitate of Hg(NH<sub>2</sub>)Cl is formed. HgCl<sub>2</sub> + 2NH<sub>3</sub>  $\longrightarrow$  Hg(NH<sub>2</sub>)Cl  $\downarrow$  + NH<sub>4</sub>Cl (white)

 $Hg(NH_2)Cl$  is soluble in a large excess of the reagent.

#### 5. With KI solution

Red precipitate of HgI<sub>2</sub> is formed.

 $HgCl_2 + 2KI \longrightarrow HgI_2 \downarrow + 2KCl$  (red)

HgI<sub>2</sub> is soluble in excess of the reagent.

 $HgI_2 + 2KI \longleftarrow K_2 [HgI_4]$ 

# Reactions of the Bismuth ion, Bi<sup>3+</sup>

1. With sulphide ion (use H<sub>2</sub>S)

0

Brown precipitate of Bi<sub>2</sub>S<sub>3</sub> is formed.

 $Bi(NO_3)_3 + 3H_2S \longrightarrow Bi_2S_3 + 6HNO_3$ (brown)

 $Bi_2S_3$  is insoluble in cold dilute acids, but soluble in hot dilute HNO<sub>3</sub> and in boiling concentrated HCl

 $Bi_2S_3 + 2NO_3 + 8H_3O^+ \longrightarrow 2Bi^{3+} + 2NO + 3S + 12H_2O$ 

#### 2. With NaOH solution

White precipitate of Bi(OH)<sub>3</sub> is formed.

 $Bi(NO_3)_3 + 2NaOH \longrightarrow Bi(OH)_3 \downarrow + 3NaNO_3$ (white)

Bi(OH)<sub>3</sub> becomes yellow on boiling, due to dehydration.

 $Bi(OH)_3 \longrightarrow BiO.OH \downarrow + H_2O$ (yellow)

#### 3. With ammonia solution

Deep blue solution of  $(OH)_3$  is formed.

$$Bi(NO_3)_3 + 3NH_4OH \longrightarrow Bi(OH)_3 \downarrow + 3NH_4NO_3$$
(white)

Bi(OH)<sub>3</sub> is insoluble in excess of the reagent.

4. With KI solution

Dark brown precipitate of Bil<sub>3</sub> is formed.

 $Bi(NO_3)_3 + 3KI \longrightarrow BiI_3 \downarrow + 3KNO_3$ (brown)

BiI<sub>3</sub> is soluble in excess of the reagent to give a yellow solution of  $K[BiI_4]$ .

 $BiI_3 + KI \longrightarrow K[BiI_4]$ 

K[BiI<sub>4</sub>] is decomposed upon dilution giving an orange–coloured precipitate of (BiO)I.

 $BiI_3 + H_2O \longrightarrow 2HI + (BiO)I$ 

## 5. With sodium stannite solution

Black precipitate of finely divided Bi is formed.

Sodium stannite Na<sub>2</sub>[SnO<sub>2</sub>] is freshly prepared by adding 3M NaOH solution drop by drop to a solution of stannous chloride

until the initial white precipitate of Sn(OH)<sub>2</sub> just dissolves.

 $Sn(OH)_{2} + 2NaOH \implies Na_{2}[SnO_{2}] + 2H_{2}O$   $2Bi(NO_{3})_{3} + 6NaOH + 3Na_{2}[SnO_{2}] \implies$   $2Bi + 3Na_{2}[SnO_{3}] + 6NaNO_{3} + 3H_{2}O$ (black)

# Reactions of the Cupric ion, Cu<sup>2+</sup>

#### 1. With sulphide ion (use $H_2S$ )

Black precipitate of CuS is formed.

 $CuSO_4 + H_2S \longrightarrow CuS + H_2SO_4$ (black)

CuS is insoluble in NaOH solution, but is soluble in hot dilute HNO<sub>3</sub> and in KCN solution.

 $3CuS + 8HNO_3 \longrightarrow 3Cu(NO_3)_2 + 3S + 4H_2O + 2NO$ 

#### 2. With NaOH solution

Blue precipitate of Cu(OH)<sub>2</sub> is formed.

 $CuSO_4 + 2NaOH \leftarrow Cu(OH)_2 \downarrow + Na_2SO_4$ (blue)

Cu(OH)<sub>2</sub> is converted on boiling into black precipitate of CuO.

 $Cu(OH)_2 \longrightarrow CuO + H_2O$ (black)

#### 3. With ammonia solution

Deep blue solution of  $[Cu(NH_3)_4]SO_4$  is formed.

 $2CuSO_4 + 8NH_3 + 2H_2O \longrightarrow 2[Cu(NH_3)_4]SO_4 + 2H_2O$ 

#### 4. With potassium ferrocyanide K<sub>4</sub>[Fe(CN)<sub>6</sub>] solution

Reddish-brown precipitate of  $Cu_2[Fe(CN)_6]$  is formed.  $CuSO_4 + K_4[Fe(CN)_6] \longrightarrow Cu_2[Fe(CN)_6] \checkmark + 2K_2SO_4$ (reddish-brown)

 $Cu_2[Fe(CN)_6]$  is insoluble in dilute acids, but dissolves in aqueous ammonia forming a blue solution.

5. With KI solution

Yellowish-brown mixture of  $Cu_2I_2$  (white) and  $I_2$  (yellowishbrown) precipitate is formed.

 $2CuSO_4 + 4KI \qquad \longleftarrow \qquad Cu_2I_2 \bigvee + I_2 \bigvee + 2K_2SO_4 \\ (white) \qquad (yellowish-brown)$ 

# Reactions of the Cadmium ion, Cd<sup>2+</sup>

#### 1. With sulphide ion (use $H_2S$ )

Yellow precipitate of CdS is formed in a neutral or a slightly acidic solution.

 $CdSO_4 + H_2S \longrightarrow CdS + H_2SO_4$ (yellow)

CdS is insoluble in KCN solution, but is soluble in hot dilute nitric acid.

#### 2. With NaOH solution

White precipitate of Cd(OH)<sub>2</sub> is formed.

 $CdSO_4 + 2NaOH \longrightarrow Cd(OH)_2 \downarrow + Na_2SO_4$ (white)

 $Cd(OH)_2$  is insoluble in excess of the reagent.

### 3. With ammonia solution

3

White precipitate of  $Cd(OH)_2$  is formed.  $CdSO_4 + 2NH_3 + 2H_2O \longrightarrow Cd(OH)_2 \downarrow^+ (NH_4)_2SO_4$ (white)

 $Cd(OH)_2$  is soluble in excess of the reagent due to the formation of  $[Cd(NH_3)_4]SO_4$ .

 $Cd(OH)_2 + (NH_4)_2SO_4 + 2NH_3$ 



اهمد الحاج ماجد العبدربه ترطاسية - طباعة - استنساح - رونيو سامراء - القاطول - ٧٢٠٧٣٦