Al-Karkh University of Science
College of science
First year level
General chemistry Labs

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Lab -9-

Separation of I Group cations (Ag^+, Pb^{2+}, Hg_2^{2+})

1. Introduction:

Group I cations (Ag^+, Hg_2^{2+}, Pb^{2+}) form insoluble chlorides. Upon the addition of hydrochloric acid Ag^+, Pb^{2+}, Hg_2^{2+} ions will precipitate as AgCl, $PbCl_2$ and Hg_2Cl_2

$AgNO_3 + HCl \rightarrow AgCl\downarrow + HNO_3$ (reaction 1)
$Pb(NO_3)_2 + 2\,HCl \rightarrow PbCl_2\downarrow + 2\,HNO_3$ (reaction 2)
$Hg_2(NO_3)_2 + 2\,HCl \rightarrow Hg_2Cl_2\downarrow + 2\,HNO_3$ (reaction 3)

The solubility of $PbCl_2$ increases approximately threefold as the temperature of the solution increases from 20°C to 100°C. Thus, $PbCl_2$ will dissolve in hot water while AgCl and Hg_2Cl_2 remains insoluble. The presence of Pb^{2+} ions in obtained solution can be proved with KI, K_2CrO_4 and H_2SO_4 solutions. Their addition yield a golden yellow precipitates of PbI_2 and $PbCrO_4$ (reactions 4 and 5), and white precipitate of $PbSO_4$ (reaction 6).

$Pb^{2+} + 2\,KI \rightarrow PbI_2\downarrow + 2\,K^+$ (reaction 4)
$Pb^{2+} + K_2CrO_4 \rightarrow PbCrO_4\downarrow + 2\,K^+$ (reaction 5)
$Pb^{2+} + H_2SO_4 \rightarrow PbSO_4\downarrow + 2\,H^+$ (reaction 6)

The precipitate still may contain Hg_2Cl_2 and AgCl. Of those two compounds, only the silver chloride is soluble in aqueous ammonia due to the formation a colorless solution of $Ag(NH_3)_2Cl$ (reaction 7), whereas mercury(I) chloride turns into Hg metal and $HgNH_2Cl$ visible as grayish-black precipitate which is insoluble in ammonia solution (reaction 8)

$AgCl\downarrow + 2\,NH_3 \rightarrow Ag(NH_3)_2Cl$ (reaction 7)
$Hg_2Cl_2\downarrow + 2\,NH_3 \rightarrow Hg\downarrow + HgNH_2Cl\downarrow + NH_4Cl$ (reaction 8)

The formation of white precipitate of AgCl in reaction of $Ag(NH_3)_2Cl$ with diluted HNO_3 proves the presence of silver (reaction 9). The additional confirmation of silver ions is yellowish precipitate of silver iodide AgI obtained in the reaction with potassium iodide KI (reaction 10).

$Ag(NH_3)_2Cl + 2\,HNO_3 \rightarrow AgCl\downarrow + 2\,NH_4NO_3$ (reaction 9)
$Ag(NH_3)_2Cl + KI \rightarrow AgI\downarrow + 2\,NH_3 + KCl$ (reaction 10)

2. Procedure:

Take 2 ml of unknown for identification and add drops of 6M HCl. You should obtain white precipitate (**Precipitate A**).

Transfer the precipitate to a small beaker, and boil with 1-5 ml water. Filter hot.

Residue: $AgCl, Hg_2Cl_2$		**Filtrate: $PbCl_2$**
Wash the ppt. with hot water. Add NH_4OH to the precipitate and filter.		
Residue: black precipitate	**Solution: Ag^+ complex**	**Cool a portion of the solution: a white crystalline ppt. of $PbCl_2$ is obtained if Pb is present in any quantity.**
$HgNH_2Cl + Hg$	**$[Ag(NH_3)_2]^+$**	
Hg_2^{2+}	**Ag^+**	**Pb^{2+}**

Additional reactions for identification:

	Divide the filtrate into two parts: **a.) Acidify with dilute HNO_3. White ppt. of AgCl.** **b.) Add a few drops of KI solution. Pale yellow ppt. of AgI.** **Ag^+ present.**	**Divide the hot filtrate into three parts:** **a.) Add K_2CrO_4 solution. Yellow ppt. of $PbCrO_4$, insoluble in dilute acetic acid.** **b.) Add KI solution. Yellow ppt. of PbI_2, soluble in hot water to a colourless solution, which deposites brilliant yellow crystals upon cooling.** **c.) Add dilute H_2SO_4. White ppt. of $PbSO_4$, soluble in ammonium tartrate solution.** **Pb^{2+} present.**

3. Results:

4. Discuss:

a- $PbCl_2$ soluble in hot water.

b- Presence of Silver ion (Ag^+) in the unknown solution.

c- Divide the colorless filtrate solution of $Ag(NH_3)_2Cl$ into two parts.

Take 2 ml of unknown for identification and add drops of 6M HCl. You should obtain white precipitate (**Precipitate A**).
Transfer the precipitate to a small beaker, and boil with 1-5 ml water. Filter hot.

Residue: $AgCl$, Hg_2Cl_2		**Filtrate: $PbCl_2$**
Wash the ppt. with hot water. Add NH_4OH to the precipitate and filter.		
Residue: black precipitate $HgNH_2Cl + Hg$	**Solution: Ag^+ complex** $[Ag(NH_3)_2]^+$	**Cool a portion of the solution: a white crystalline ppt. of $PbCl_2$ is obtained if Pb is present in any quantity.**
Hg_2^{2+}	Ag^+	Pb^{2+}

Additional reactions for identification:

	Divide the filtrate into two parts: **a.) Acidify with dilute HNO_3. White ppt. of AgCl.** **b.) Add a few drops of KI solution. Pale yellow ppt. of AgI.** **Ag^+ present.**	**Divide the hot filtrate into three parts:** **a.) Add K_2CrO_4 solution. Yellow ppt. of $PbCrO_4$, insoluble in dilute acetic acid.** **b.) Add KI solution. Yellow ppt. of PbI_2, soluble in hot water to a colourless solution, which deposites brilliant yellow crystals upon cooling.** **c.) Add dilute H_2SO_4. White ppt. of $PbSO_4$, soluble in ammonium tartrate solution.** **Pb^{2+} present.**

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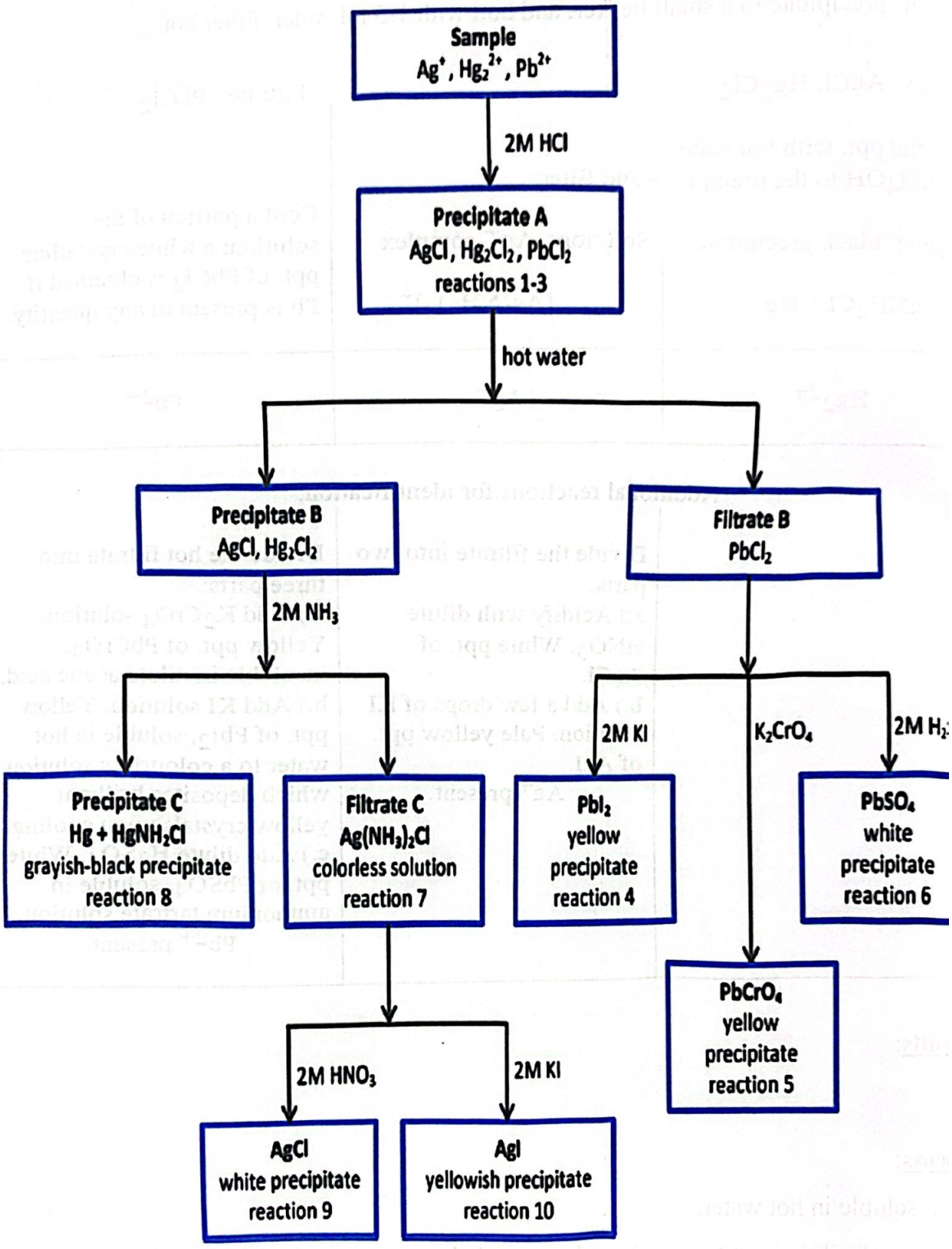

Scheme 1. Analysis of the Group I.