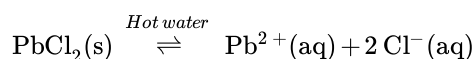


## 3.2: Separation and confirmation of individual ions in group I precipitates

### Separation and confirmation of lead(II) ion

Solubility of  $\text{PbCl}_2$  in water at 20 °C is about 1.1 g/100 mL, which is significantly higher than  $1.9 \times 10^{-4}$  g/100 mL for  $\text{AgCl}$  and  $3.2 \times 10^{-5}$  g/100 mL for  $\text{Hg}_2\text{Cl}_2$ . Further, the solubility of  $\text{PbCl}_2$  increases three-fold to about 3.2 g/100 mL in boiling water at 100 °C, while solubility  $\text{AgCl}$  and  $\text{Hg}_2\text{Cl}_2$  remain negligible. A 15 drops sample that is used to precipitate out group I cations corresponds to about 0.75 mL, which based on the molar mass of  $\text{PbCl}_2$  is 278.1 g and the concentration of each ion ~0.1M, contains about 0.02 g of  $\text{PbCl}_2$  precipitate. This 0.02 g of  $\text{PbCl}_2$  requires ~0.6 mL of heated water for dissolution. The precipitated is re-suspended in ~2 mL water and heated in a boiling water bath to selectively dissolve  $\text{PbCl}_2$ , leaving any  $\text{AgCl}$  and  $\text{Hg}_2\text{Cl}_2$  almost undissolved, as shown in Figure 3.2.1.



The heated suspension is filtered using a heated gravity filtration set up to separate the residue comprising of  $\text{AgCl}$  and  $\text{Hg}_2\text{Cl}_2$  from filtrate containing dissolved  $\text{PbCl}_2$ .

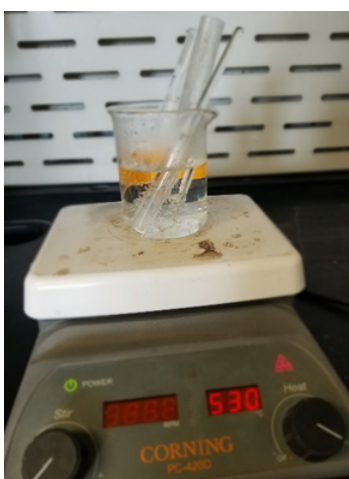
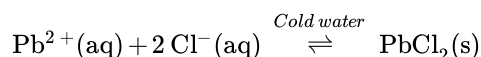


Figure 3.2.1: Group I precipitate resuspended in water and being heated in a water bath to redissolve  $\text{PbCl}_2$ .

The solubility of  $\text{PbCl}_2$  is three times less at room temperature than in boiling water. Therefore, the 2 mL filtrate is cooled to room temperature to crystalize out  $\text{PbCl}_2$  :



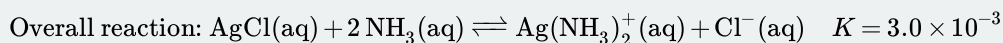
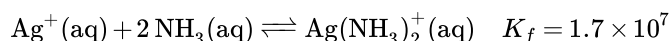
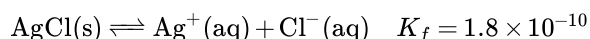
If  $\text{PbCl}_2$  crystals are observed in the filtrate upon cooling to room temperature, it is a confirmation of  $\text{PbCl}_2$  in the test solution. If  $\text{PbCl}_2$  concentration is low in the filtrate, the crystals may not form upon cooling. Few drops of 5M  $\text{HCl}$  are mixed with the filtrate to force the crystal formation based on the **common ion effect** of  $\text{Cl}^{-}$  in the reactants. The formation of  $\text{PbCl}_2$  crystals confirms  $\text{Pb}^{2+}$  as shown in Figure 3.2.2, and no crystal formation at this stage confirms that  $\text{Pb}^{2+}$  was absent in the test solution.



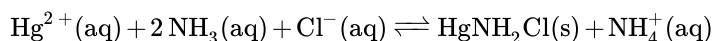
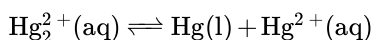
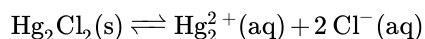
Figure 3.2.2:  $\text{PbCl}_2$  recrystallized upon cooling the hot solution and addition of two drops of 6M HCl.

### Separating mercury(I) ion from silver(I) ion and confirming mercury(I) ion

The residue left after filtering out  $\text{Pb}^{2+}$  in hot water, is washed further with 10 mL of hot water to washout residual  $\text{PbCl}_2$ . Then 2 mL of 6M aqueous  $\text{NH}_3$  solution is passed through the residue drop by drop. Aqueous  $\text{NH}_3$  dissolves  $\text{AgCl}$  precipitate by forming water soluble complex ion  $[\text{Ag}(\text{NH}_3)_2(\text{aq})]^+$  through following series of reactions:



The 2 mL filtrate is collected in a separate test tube for confirmation of  $\text{Ag}^+$  ion. Although  $\text{Hg}_2\text{Cl}_2$  precipitate is insoluble in water, it does slightly dissociate like all ionic compounds. The  $\text{Hg}_2^{2+}$  ions undergo auto-oxidation or disproportionation reaction producing black Hg liquid and  $\text{Hg}_2^{2+}$  ions. The  $\text{Hg}_2^{2+}$  ions react with  $\text{NH}_3$  and  $\text{Cl}^-$  forming white water-insoluble  $\text{HgNH}_2\text{Cl}$  precipitate through the following series of reactions:



A mixture of white solid  $\text{HgNH}_2\text{Cl}$  and black liquid Hg appears gray in color. Turning of white  $\text{Hg}_2\text{Cl}_2$  precipitate to grayish color upon addition of 6M  $\text{NH}_3$  solution drops confirms  $\text{Hg}_2^{2+}$  ions are present in the test solution as shown in Figure 3.2.3. If the white precipitate redissolves leaving behind no grayish residue, it means the precipitate was  $\text{AgCl}$  and  $\text{Hg}_2^{2+}$  were absent in the test solution.

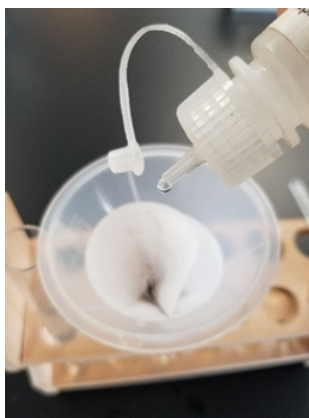
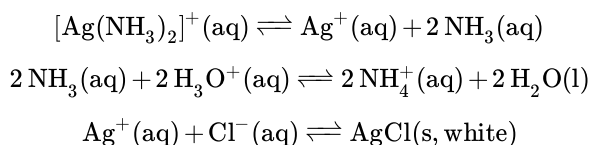


Figure 3.2.3: Confirmation of  $\text{Hg}_2^{2+}$ , i.e., white precipitates turn grayish-black upon addition of 6M  $\text{NH}_3$  solution.

### Confirming silver(I) ion

Although water-soluble complex ion  $[\text{Ag}(\text{NH}_3)_2(\text{aq})]^+$  is quite stable, it does slightly decompose into  $\text{Ag}^+$  and  $\text{NH}_3(\text{aq})$ . The excess  $\text{NH}_3$  added to dissolve  $\text{AgCl}$  precipitate and the that produced by dissociation of  $[\text{Ag}(\text{NH}_3)_2(\text{aq})]^+$  is removed by making the solution acidic by adding 6M  $\text{HNO}_3$ . The  $\text{Cl}^-$  formed from the dissolution of  $\text{AgCl}$  precipitate in the earlier reactions is still present in the medium. Decomposition of  $[\text{Ag}(\text{NH}_3)_2(\text{aq})]^+$  in the acidic medium produces enough  $\text{Ag}^+$  ions to re-form white  $\text{AgCl}$  precipitate by the following series of equilibrium reactions.



Overall reaction:  $[\text{Ag}(\text{NH}_3)_2]^+(\text{aq}) + 2\text{H}_3\text{O}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightleftharpoons \text{AgCl}(\text{s, white}) + 2\text{NH}_4^+(\text{aq}) + 2\text{H}_2\text{O}(\text{l})$

The formation of white  $\text{AgCl}$  precipitate at this stage in the acidified filtrate confirms  $\text{Ag}^+$  ion was present in the test solution, as shown in Figure 3.2.4, and its absence confirms that  $\text{Ag}^+$  ion was not present in the test solution.



Figure 3.2.4: Confirmation of  $\text{Ag}^+$ : formation of white precipitate upon converting  $[\text{Ag}(\text{NH}_3)_2(\text{aq})]^+$  solution from alkaline to acidic by addition of  $\text{HNO}_3$ .

This page titled [3.2: Separation and confirmation of individual ions in group I precipitates](#) is shared under a [Public Domain](#) license and was authored, remixed, and/or curated by [Muhammad Arif Malik](#).

- [Current page](#) by [Muhammad Arif Malik](#) is licensed [Public Domain](#).
- [1.3: Varying solubility of ionic compounds](#) by [Muhammad Arif Malik](#) is licensed [Public Domain](#).