

# Lab: Qualitative Analysis of an Inorganic Compound

*Prelab - This must be done before you can start your testing.*

Read p. 809-825 (17.5-17.7) ---Put this in your lab notebook; the foldable can be glued)

Part A. Explain the meaning of the following: qualitative analysis, quantitative analysis, complexation, decanting, solubility product, ligand, coordinate covalent bond, centrifuging, selective precipitation

Part B. Go to: <http://web.mst.edu/~gbert/qual/qual.html> and click on "Discussion." Read through the background information as you answer the questions below.

1. What is  $K_{sp}$  and what does it have to do with forming a precipitate?
2. How are the cations grouped for testing?
3. How is LeChatelier's principle used in qualitative analysis?
4. From the above website's "Discussion," do the online "suggested experiment." Write your observations here in your lab notebook by this prelab question. Start by clicking on the lab reagent bottle and the click on a TT and click to put drops in. By following the procedure her you'll get familiar with the qualitative analysis of Part I cations. Do a trial unknown and check your results. Did you solve it correctly?

## Cation Testing of Ionic Compounds

### Background Information:

Qualitative analysis is a type of analysis that informs you of the presence of a substance, but does not tell you exactly how much of the substance there is. It is like being able to get a YES or NO answer to the question "is a particular metal ion present in this sample?" Cations are analyzed by the careful separation (precipitation) reactions and anions can be analyzed by initial solubility with compounds and then individual tests. What are the ways in which ions can be removed from a solution and thus drive a METATHESIS reaction? Certain ions can associate to form an insoluble precipitate. Precipitation reactions: *Solubility refers to the amount of a substance that can be dissolved in a given quantity of water. This is a consideration of the relative strength of water-ion vs. Ion-ion attractive forces. If the ion-ion attractive forces > water-ion attractive forces, then the ion-ion interaction will predominate, resulting in precipitation (lack of solvation). A solubility less than 0.01 M will be considered as essentially insoluble.*

Common Qualitative Analysis Reagents	Reagent Effects
6M HCl	Increases $[H^+]$ Increases $[Cl^-]$ Decreases $[OH^-]$ Dissolves insoluble carbonates, chromates,

	hydroxides, some sulfates Destroys hydroxo and NH <sub>3</sub> complexes Precipitates insoluble chlorides
6M HNO <sub>3</sub>	Increases [H <sup>+</sup> ] Decreases [OH <sup>-</sup> ] Dissolves insoluble carbonates, chromates, and hydroxides Dissolves insoluble sulfides by oxidizing sulfide ion Destroys hydroxo and ammonia complexes Good oxidizing agent when hot
6M NaOH	Increases [OH <sup>-</sup> ] Decreases [H <sup>+</sup> ] Forms hydroxo complexes Precipitates insoluble hydroxides
6M NH <sub>3</sub>	Increases [NH <sub>3</sub> ] Increases [OH <sup>-</sup> ] Decreases [H <sup>+</sup> ] Precipitates insoluble hydroxides Forms NH <sub>3</sub> complexes Forms a basic buffer with NH <sub>4</sub> <sup>+</sup>

Among the most common reactions in qualitative analysis are those involving the formation or decomposition of complex ions and precipitation reactions. These reactions may be performed directly by adding the appropriate anion, or a reagent such as H<sub>2</sub>S or NH<sub>3</sub> may dissociate in water to furnish the anion. Strong acid may be used to dissolve precipitates containing a basic anion. Ammonia or sodium hydroxide may be used to bring a solid into solution if the cation in the precipitate forms a stable complex with NH<sub>3</sub> or OH<sup>-</sup>.

#### Complexes of cations with NH<sub>3</sub> and OH<sup>-</sup>

Cation	NH <sub>3</sub> Complex	OH <sup>-</sup> Complex
Ag <sup>+</sup>	Ag(NH <sub>3</sub> ) <sub>2</sub> <sup>+</sup>	--
Al <sup>3+</sup>	--	Al(OH) <sub>4</sub> <sup>-</sup>
Cd <sup>2+</sup>	Cd(NH <sub>3</sub> ) <sub>4</sub> <sup>2+</sup>	--
Cu <sup>2+</sup>	Cu(NH <sub>3</sub> ) <sub>4</sub> <sup>2+</sup> (blue)	--
Ni <sup>2+</sup>	Ni(NH <sub>3</sub> ) <sub>6</sub> <sup>2+</sup> (blue)	--

$\text{Pb}^{2+}$	--	$\text{Pb}(\text{OH})_3^-$
$\text{Sb}^{3+}$	--	$\text{Sb}(\text{OH})_4^-$
$\text{Sn}^{4+}$	--	$\text{Sn}(\text{OH})_6^{2-}$
$\text{Zn}^{2+}$	$\text{Zn}(\text{NH}_3)_4^{2+}$	$\text{Zn}(\text{OH})_4^{2-}$

A cation is usually present as a single principal species, which may be a complex ion, free ion, or precipitate. If the reaction goes to completion the principal species is a complex ion. The precipitate is the principal species if most of the precipitate remains undissolved. If a cation forms a stable complex, addition of a complexing agent at 1 M or greater generally will convert the free ion to complex ion.

The dissociation constant  $K_d$  can be used to determine the extent to which a cation is converted to a complex ion. The solubility product constant  $K_{sp}$  can be used to determine the fraction of cation remaining in a solution after precipitation.  $K_d$  and  $K_{sp}$  are both required to calculate the equilibrium constant for dissolving a precipitate in a complexing agent.

### HINTS FOR ALL PARTS:

1ml = 20 drops

$$K_{sp} = [A^{+}(\text{aq})][X^{-}(\text{aq})]$$

If the solubility is 0.01 M, then  $[A^{+}(\text{aq})] = 0.01 \text{ M}$  and  $[X^{-}(\text{aq})] = 0.01 \text{ M}$ , thus:

$$K_{sp} = (0.01)(0.01) = 1.0 \times 10^{-4}$$

Generally speaking, if  $K_{sp} < 1.0 \times 10^{-4}$ , the ionic compound is considered to be insoluble

### Part I: $\text{Ag}^{+1}$ , $\text{Hg}^{+2}$ , $\text{Pb}^{+2}$

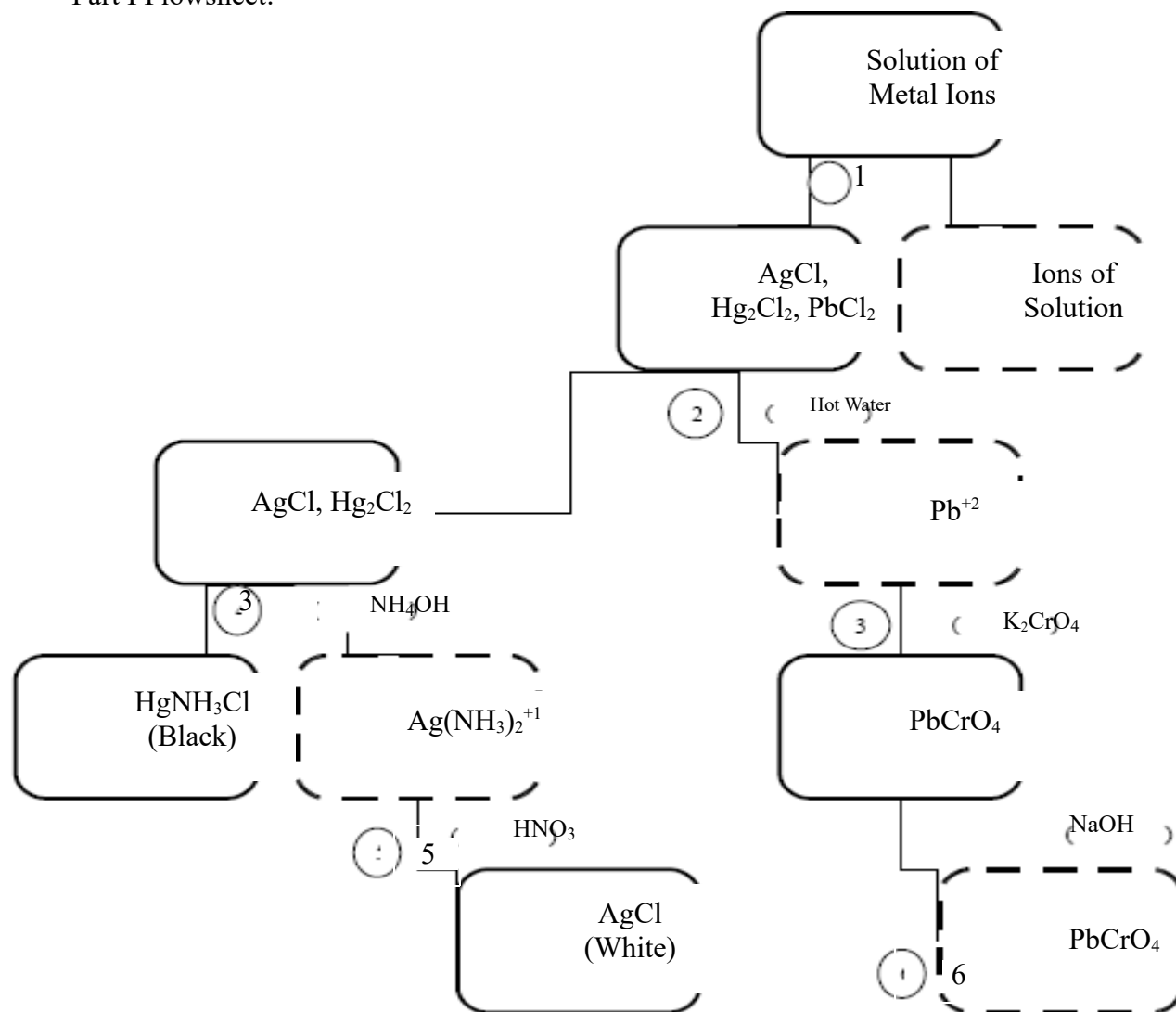
Procedure:

Prepare a KNOWN sample by placing approximately 1 ml of nitrate salt solution of each of the ions above into the SAME test tube.

1. For the solution to be tested, add 6 M HCl drop by drop until any precipitation that occurs is complete. AVOID a large excess of HCl. Filter into a test tube and test the filtrate for complete filtration with one drop of HCl. If precipitation occurs, repeat the above procedure until precipitation is one drop of HCl. If precipitation occurs, repeat the above procedure until precipitation is complete. Wash the precipitate on the filter paper by spraying with 1 – 3 ml of DI water from your wash bottle. (When doing the general unknown, add the wash water to the filtrate and save for Part II).
2. Heat 10 - 15 ml of water to boiling, and then pour 3 – 4 ml of this boiling water over the precipitate on the filter paper, catching the hot filtrate in a TT. We perform this because  $\text{PbCl}_2$  is far more soluble than the chlorides of the other two ions, especially in hot water. Wash the remaining residue on the filter paper by pouring 10 ml of boiling water over it in portions.
3. Add several drops of 1.0 M  $\text{K}_2\text{CrO}_4$  to the filtrate. A yellow precipitate proves  $\text{Pb}^{+2}$ . This may be confirmed by added 6 M NaOH to dissolve the yellow precipitate. You may not need to dissolve it all.
4. To the residue from the hot-water treatment, on the filter paper add 1 ml of 6 M  $\text{NH}_3(\text{aq})$  and then add 2 ml of water, collection the filtrate in a test tube. A black residue on the filter paper proves  $\text{Hg}_2^{+2}$ .
5. If the above  $\text{NH}_3(\text{aq})$  filtrate is not perfectly clear, colloidal  $\text{Pb}(\text{OH})\text{Cl}$  may be coming through the filter paper. If this happens, re-filter the solution through the same filter to obtain a clean filtrate. Acidify the clear filtrate with 6 M  $\text{HNO}_3$  and mix. A white

precipitate AgCl proves the presence of  $\text{Ag}^{+1}$ . The solution MUST be acidic for the precipitate to form. If in doubt about the acidity, test the solution with hydron paper.

Part I Flowsheet:



**Data:** observations after each step and reasoning for solubility. Ppt or no ppt and why? Write net ionic equations for each step in your procedure.

**Conclusion:** Purpose? Techniques used? Principle behind the lab? Compare and contrast the procedure for online “suggested experiment” and the one done in lab.

Error?

Resources?