



# General Chemistry Laboratory

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## Qualitative Analysis of Group 2 Cations



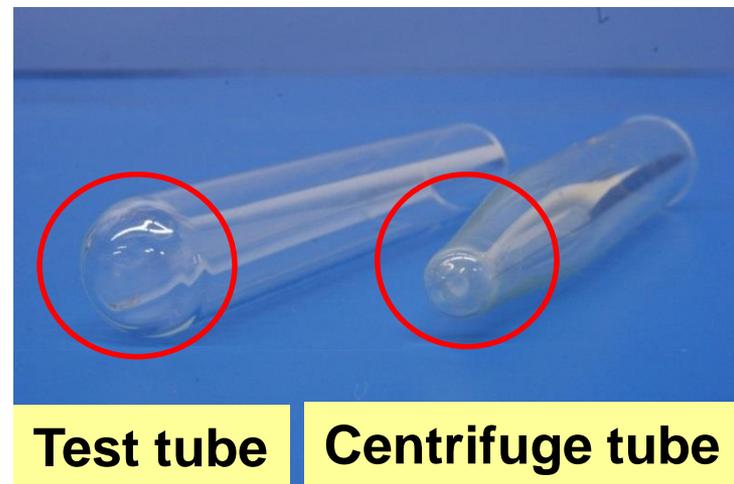
# Preparation

## Collect the following items

- Five centrifuge tubes, test tube tongs
- Dropping pipettes
- Crucible tongs
- Evaporating dish
- Sticky labels

## From your personal equipment

- Centrifuge
- Test tube rack, test tubes, and beaker



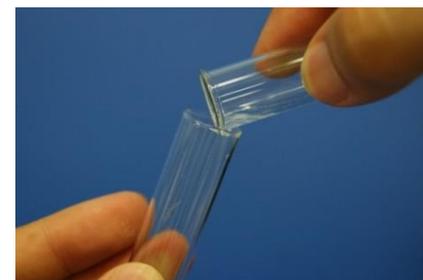


# Objective and Principles

- **Objective:** Separate and identify common Group 2 cations from a mixture solution based on the concepts of precipitation, dissolution, and formation of complex ions
- **Lab techniques:**
  - Systematic analysis of cations
  - Vortex mixer
  - Operating a centrifuge
  - Decantation
  - Using litmus paper to determine pH



**Vortex mixer**

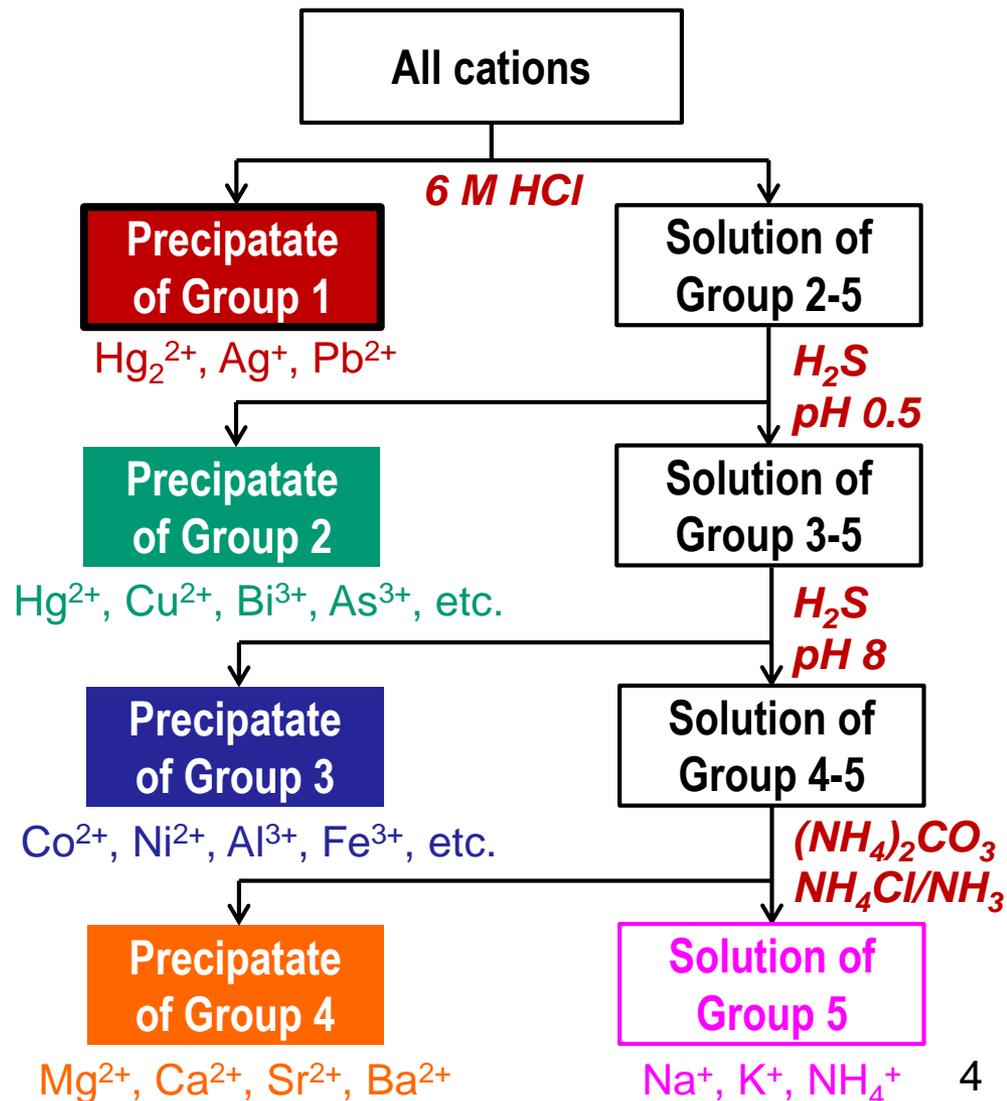


**Decantation**



# Qualitative Analysis of Cations

- I. Separating cations into five groups based on their solubilities in the presence of various precipitating reagents
- II. Selective and sequential dissolution of cations in the same group
- III. Verifying individual cations





# Qualitative Analysis of Cations

## Cationic Solutions

- (I) Insoluble chlorides:  $\text{Hg}_2^{2+}$ ,  $\text{Ag}^+$ ,  $\text{Pb}^{2+}$
- (II) Insoluble sulfides in acidic medium:  $\text{Hg}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Cd}^{2+}$ ,  $\text{As}^{3+}$ ,  $\text{Sb}^{3+}$ ,  $\text{Sn}^{4+}$  (**metallic sulfide precipitates with smaller  $K_{sp}$** )
- (III) Insoluble sulfide or hydroxides in alkaline medium:  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Mn}^{2+}$  (**metallic sulfide precipitates with greater  $K_{sp}$** )
- (IV) Insoluble Carbonates:  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$
- (V) Soluble cations:  $\text{NH}_4^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$

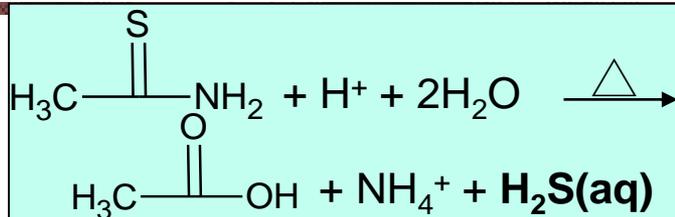


# Subgroups Of Group 2 Cations

- Group 2 cations form insoluble sulfides in acidic medium i.e.  $\text{HgS}$ ,  $\text{PbS}$ ,  $\text{CuS}$ ,  $\text{Bi}_2\text{S}_3$ ,  $\text{CdS}$ ,  $\text{As}_2\text{S}_3$ ,  $\text{Sb}_2\text{S}_3$ ,  $\text{SnS}_2$ 
    - **Copper subgroup** –  $\text{Hg}^{2+}$ ,  $\text{Pb}^{2+}$ ,  **$\text{Cu}^{2+}$** ,  **$\text{Bi}^{3+}$** ,  $\text{Cd}^{2+}$   
The sulfides are insoluble in KOH solution, only soluble in nitric acid
    - **Arsenic subgroup** –  $\text{As}^{3+}$ ,  **$\text{Sb}^{3+}$** ,  **$\text{Sn}^{4+}$**   
The sulfides are thioamphoteric that are soluble in  $\text{KOH}(\text{aq})$  and nitric acid
- ✓ Most of group 2 cations are toxic heavy metals, thus we only examine  $\text{Cu}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Sb}^{3+}$ ,  $\text{Sn}^{4+}$



# Step 1/4: Precipitating Sulfides



- Label a centrifuge tube
- Mix  $\text{Cu}^{2+}/\text{Bi}^{3+}/\text{Sb}^{3+}/\text{Sn}^{4+}$  (2/2/2/8 drops) and add 13% TA to produce sulfides
  - Mix solution (finger-flicking, glass rod, or Vortex)
  - Heat in warm water to produce sulfide



- Centrifuge 1 min and decant the supernatant



# Step 2/4: Separate Copper and Arsenic Subgroups

Cationic solution  
 $\text{Cu}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Sb}^{3+}$ ,  $\text{Sn}^{4+}$   
(2, 2, 2, 8 drops)

(pH 0.5)  
2 d 13%TA,  $\Delta$   
Centrifuge and separate  
(Repeat 13%TA precipitation once)

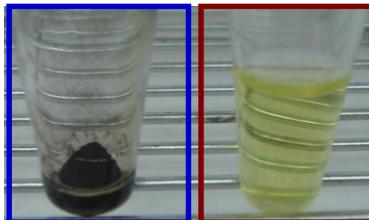


**Ppt 2-1**  
 $\text{Bi}_2\text{S}_3$ ,  $\text{CuS}$ ,  $\text{Sb}_2\text{S}_3$ ,  $\text{SnS}_2$

**Soln 2-1**

Wash ppt with 1 d 6 M  $\text{NH}_4\text{Cl}$  and 20 d of water, cfg.  
Add 10 d 0.5 M **KOH** to ppt and mix well  
Heat in boiling water bath  
Cfg. and separate the ppt and supernatant  
(Repeat this extraction with **KOH** once)

**Ppt 2-2 Copper subgroup**  
 $\text{Bi}_2\text{S}_3$ ,  $\text{CuS}$



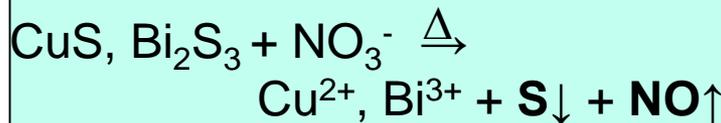
**Soln 2-2 Arsenic subgroup**  
 $\text{SbS}_3^{3-}$ ,  $\text{SbO}_3^{3-}$ ,  $\text{SnS}_3^{2-}$ ,  $\text{SnS}_2\text{OH}^-$ , (KOH)



# Step 3.1/4: Identifying $\text{Cu}^{2+}$ and $\text{Bi}^{3+}$



**Ppt 2-2 Copper subgroup**  
 **$\text{Bi}_2\text{S}_3$ ,  $\text{CuS}$**



Wash ppt with  $\text{NH}_4\text{NO}_3$ /water  
Cfg. to get ppt.  
Add 5 d. 6 M  $\text{HNO}_3$ / 5 d.  $\text{H}_2\text{O}$   
Heat in boiling water bath  
Cfg. and obtain soln



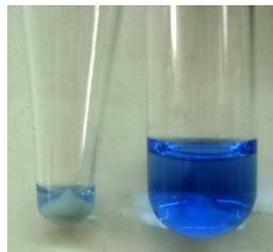
**Soln 2-3**  
 **$\text{Bi}^{3+}$ ,  $\text{Cu}^{2+}$**

Discard residue  
(Contains S)

Add and mix with **conc.  $\text{NH}_3(\text{aq})$**  until basic  
(confirm with litmus test)  
Cfg. and separate ppt and supernatant

**Ppt 2-4**  
 **$\text{Bi}(\text{OH})_3(\text{s})$  (white)**

**Soln 2-4**  
 **$\text{Cu}(\text{NH}_3)_4^{2+}$  (deep blue)**



Add  
**sodium stannite reagent**  
 $\text{Sn}(\text{OH})_3^-$  (freshly prepared)

**$\text{Bi}(\text{s})$  (black)**



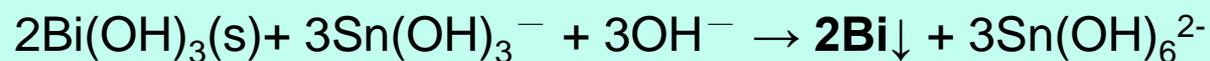


## Step 3.2/4: Prepare Sodium Stannite Reagent



**Ppt 2-4**  
**Bi(OH)<sub>3</sub>(s) (white)**

Add  
**sodium stannite reagent Sn(OH)<sub>3</sub><sup>-</sup>**

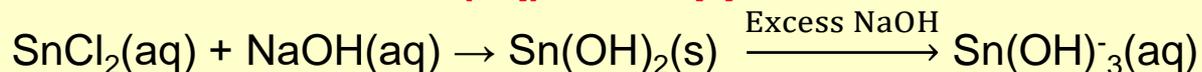


**Bi(s) (black)**



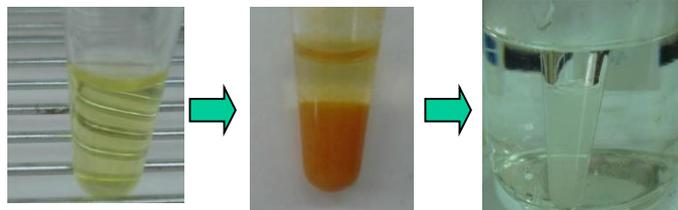
✓ Prepare sodium stannite freshly in one test tube:

✓ Add no more NaOH(aq) while ppt dissolved



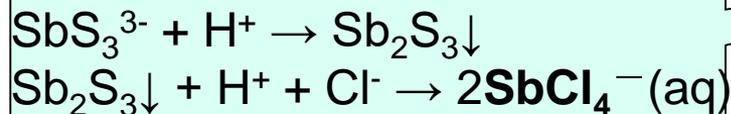


# Step 4/4: Identifying Sn<sup>4+</sup> and Sb<sup>3+</sup>



**Soln 2-2 Arsenic subgroup**  
**SbS<sub>3</sub><sup>3-</sup>, SbO<sub>3</sub><sup>3-</sup>, SnS<sub>3</sub><sup>2-</sup>, SnS<sub>2</sub>OH<sup>-</sup>**

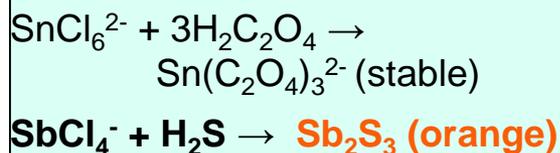
Add ca. 20 d of **conc. HCl**  
Heat in boiling water bath, till ppt dissolves  
Cfg, pour supernatant into evaporating dish



**Soln 2-6**  
**SnCl<sub>6</sub><sup>2-</sup>, SbCl<sub>4</sub><sup>-</sup>**

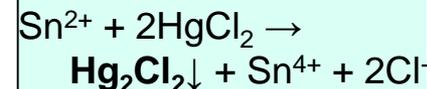
Discard  
residue

Evaporate till approx. 4 d left  
Add 1 mL water and divide into 2 parts



**SbTest**

**Sn test**

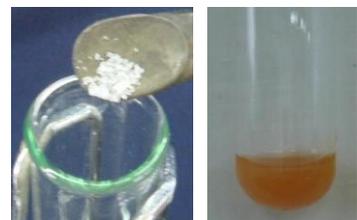


¼ small spatula **H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>(s)**  
2 d **13%TA**  
Warm in water bath

Small **AI** strip /6 M **HCl**  
Heat in boiling water bath to dissolve  
Cfg. and obtain soln  
Add 0.1 M **HgCl<sub>2</sub>** to solution

**Sb<sub>2</sub>S(s) (orange)**

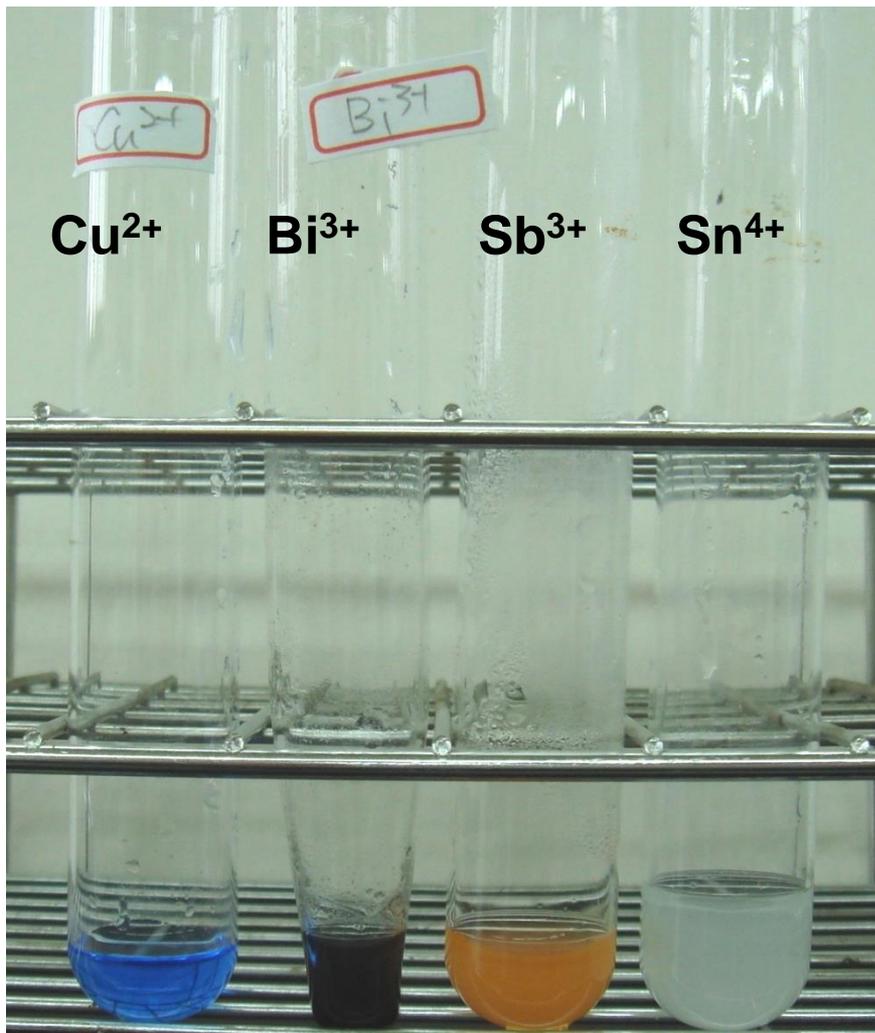
**Hg<sub>2</sub>Cl<sub>2</sub>(s) (white)**  
Hg (black)





# Record Detailed Observations

- Operations (e.g. adding x drops Y, centrifuge speed setting, repeating extraction z times, etc.)
- Reaction conditions (e.g. in fume hood, in boiling water bath, etc.)
- Phenomena (i.e. appearance of precipitates and solutions, speed of changes, etc.)
- **Present all identification products to TA at the end of lab**





# Additional Notes

- Wear NBR gloves throughout the experiment
- Use test tube rack or test tube tongs for transporting test tubes and centrifuge tubes
- Operate hot water bath, conc.  $\text{NH}_3(\text{aq})$ , and conc.  $\text{HCl}(\text{aq})$  in the fume hood
- Take only the required amount of chemicals as lab manual to minimize chemical waste

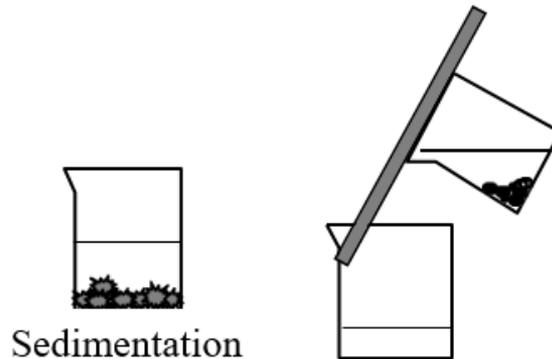


# Clean-Up and Check-Out

- All wastes should be disposed into the heavy metal recycling container
- Remove sticky labels, brush and return the centrifuge tubes
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- Tuck the lab stools underneath the lab bench
- This is a **Brief Report** experiment:
  - Member A & B: **Hand in prelab/lab note/report together to the TA**
- Groups on duty shall stay and help clean up the lab



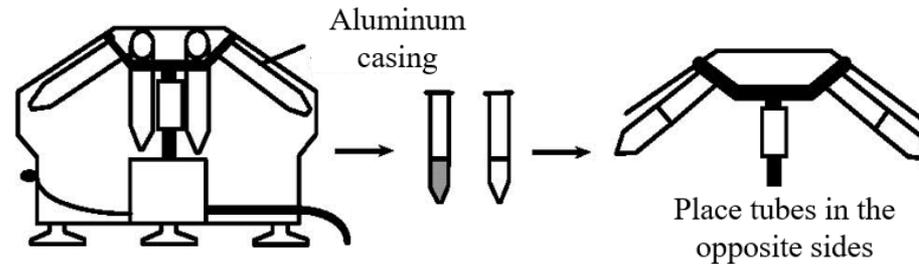
# T5 - Decantation



- Decantation is a simple method in lab to separate solids and solutions. When specific gravity of the solid precipitate is large, the solid settles quickly and tightly. When the solid settles to the bottom of the liquid and is no longer suspended in the solution, the liquid can be carefully poured out and the solid will left in the vessel and therefore separates liquid and solid.
- Stand the suspended solution by allowing the solid to settle to the bottom of the mixture
- Pour off the particle-free part of the liquid.



# T8 - Centrifugation



- Check the casing inside the machine is intact. If corrosion causes holes in casing or there is an unknown object inside, clean or replace the casing.
- Use centrifuge tubes in centrifugation, do not use test tubes.
- Centrifuge tubes should be placed in opposite sides to keep balancing.
- The lid should be closed during use; the centrifuge should be started from slow rate to check if there is any malfunction, then the speed can be increased.
- If there are unusual sounds or shaking in the centrifuge, the power should be turned off first in order to fix up.
- There must be at least one person look after the centrifuge when in use.
- Centrifuge for approx. 1~2 min, open lid when the centrifuge has completely stopped.



# T15 - Litmus Paper

- Litmus paper is filter paper which has been treated with a natural water-soluble dye obtained from lichens.
- Blue litmus paper will turn red when encountering acidic substances.
- Red litmus paper will turn blue when encountering basic substances.
- Another widely used universal indicator paper which is a combination of a variety of indicators to obtain various color changes.
- Stick solution with a clean glass rod and touch it on a litmus paper or universal indicator paper to observe the color.
- Do not dip litmus paper into solution directly to avoid contamination .
- When testing with gas, wet the litmus paper first then place it on the opening of vessel. After the gas goes out and absorbed by litmus paper, the acidity and alkalinity can be judged by color change.

