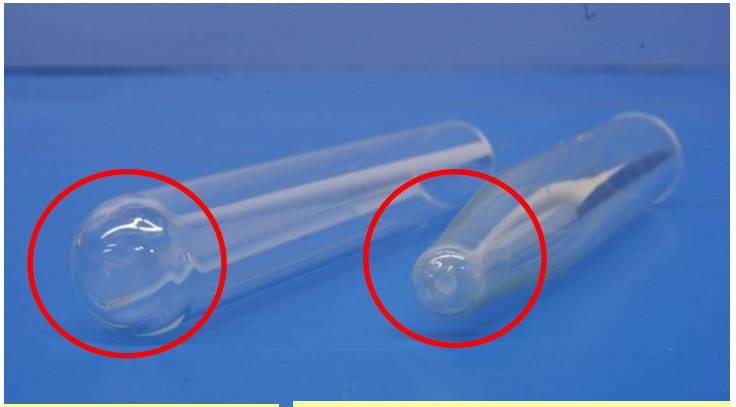
Qualitative Analysis of Group 2 Cations

Last revised: 2024/03/31

Preparation

Collect the following items

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



Test tube | Centrifuge tube

From your personal equipment

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

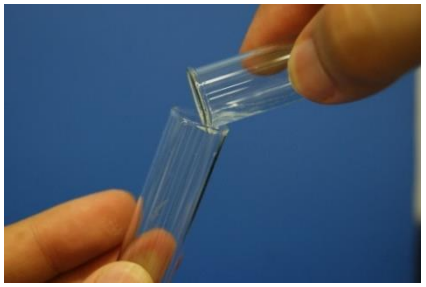
Crucible tongs

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



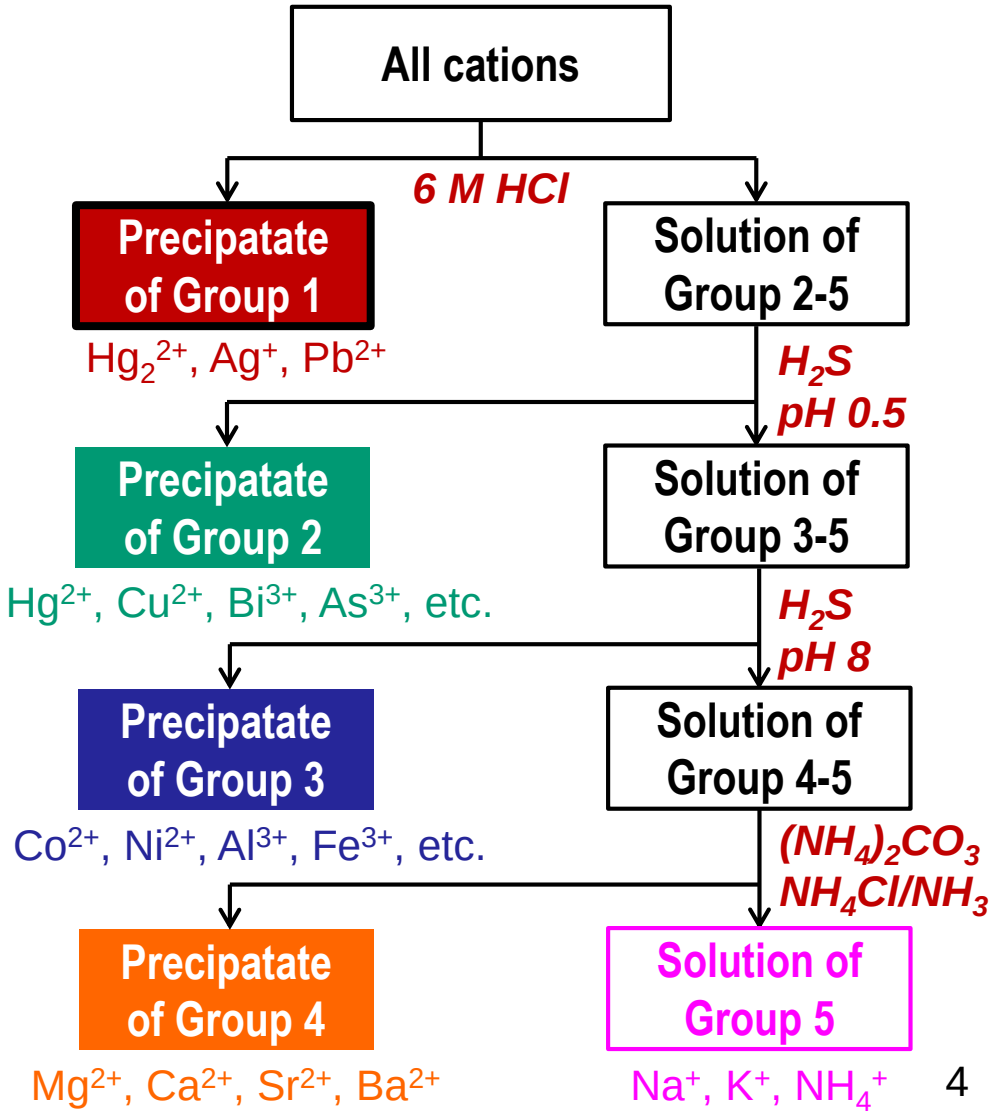
Decantation

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: Hg_2^{2+}, Ag^+, Pb^{2+}

(II) Insoluble sulfides in acidic medium: Hg^{2+}, Pb^{2+}, Cu^{2+}, Bi^{3+}, Cd^{2+}, As^{3+}, Sb^{3+}, Sn^{4+} (**metallic sulfide precipitates with smaller K_{sp}**)

(III) Insoluble sulfide or hydroxides in alkaline medium: Al^{3+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cr^{3+}, Zn^{2+}, Mn^{2+} (**metallic sulfide precipitates with greater K_{sp}**)

(IV) Insoluble Carbonates: Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}

(V) Soluble cations: NH_4^+, Na^+, K^+

Subgroups Of Group 2 Cations

- Group 2 cations form insoluble sulfides in acidic medium i.e. HgS, PbS, CuS, Bi_2S_3, CdS, As_2S_3, Sb_2S_3, SnS_2
  - **Copper subgroup** – Hg^{2+}, Pb^{2+}, **Cu^{2+}, Bi^{3+},** Cd^{2+}

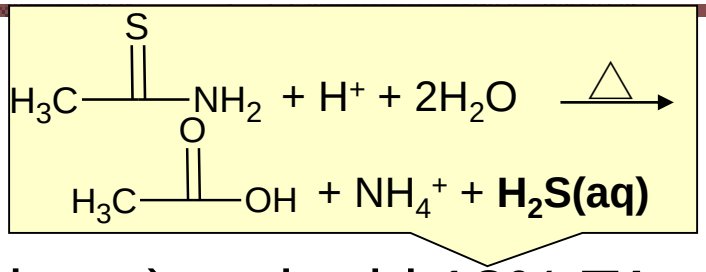
    The sulfides are insoluble in KOH solution, only soluble in nitric acid

  - **Arsenic subgroup** – As^{3+}, **Sb^{3+}, Sn^{4+}**

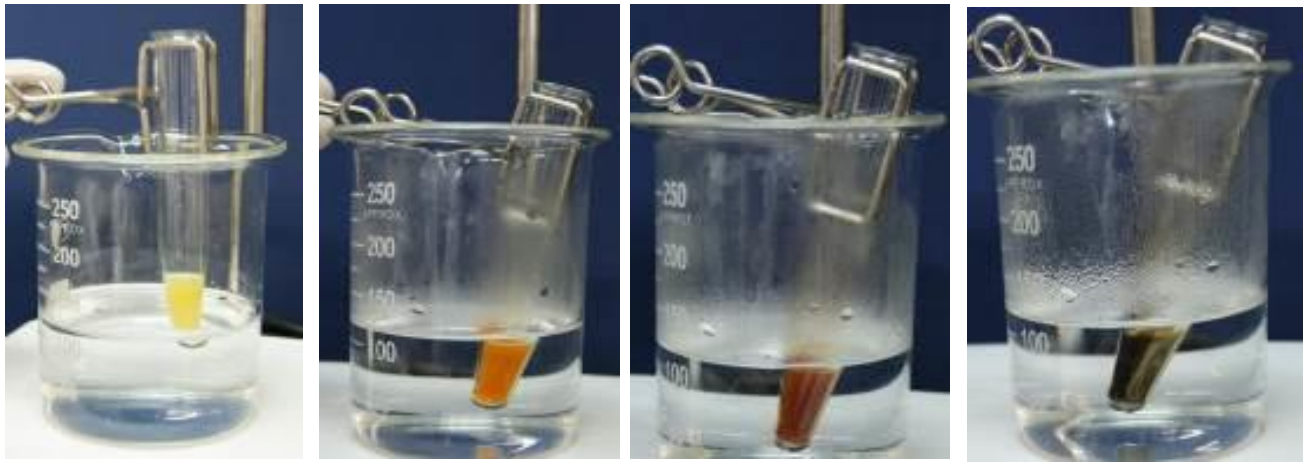
    The sulfides are thioamphoteric that are soluble in KOH(aq) and nitric acid

✓ Most of group 2 cations are toxic heavy metals, thus we only examine Cu^{2+}, Bi^{3+}, Sb^{3+}, Sn^{4+}

Step 1/4: Precipitaing Sulfides



- Label a centrifuge tube
- Mix $Cu^{2+}/Bi^{3+}/Sb^{3+}/Sn^{4+}$ (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
Cu^{2+}, Bi^{3+}, Sb^{3+}, Sn^{4+}
(2, 2, 2, 8 drops)

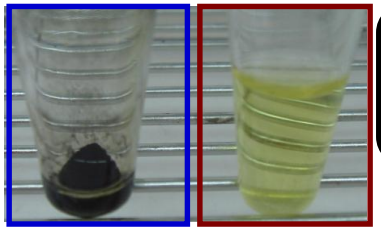
(pH 0.5)
2 d 13%TA, Δ
Centrifuge and separate
(Repeat 13%TA precipitation once)

Ppt 2-1
Bi_2S_3, CuS, Sb_2S_3, SnS_2

Soln 2-1

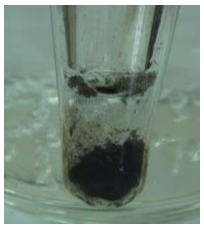
Wash ppt with 1 d 6 M NH_4Cl and 20 d of water, cfg.
Add 10 d 0.5 M **KOH** to ppt and mix well
Heat in boiling water bath
Cfg. and separate the ppt and supernatant
(Repeat this extraction with **KOH** once)

Ppt 2-2 Copper subgroup
Bi_2S_3, CuS



Soln 2-2 Arsenic subgroup
SbS_3^{3-}, SbO_3^{3-}, SnS_3^{2-}, SnS_2OH^-, (KOH)

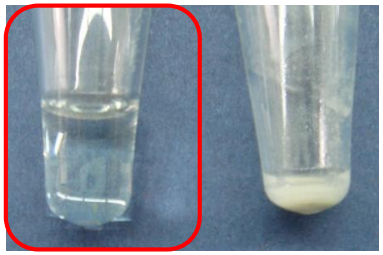
Step 3.1/4: Identifying Cu^{2+} and Bi^{3+}



Ppt 2-2 Copper subgroup
Bi_2S_3, CuS

Wash ppt with NH_4NO_3/water
Cfg. to get ppt.
Add 5 d. 6 M **HNO_3**/ 5 d. H_2O
Heat in boiling water bath
Cfg. and obtain soln

$CuS, Bi_2S_3 + NO_3^- \xrightarrow{\Delta} Cu^{2+}, Bi^{3+} + \mathbf{S}\downarrow + \mathbf{NO}\uparrow$



Soln 2-3 Bi^{3+}, Cu^{2+}

Discard residue (Contains S)

Add and mix with **conc. NH_3(aq)** until basic
(confirm with litmus test)
Cfg. and separate ppt and supernatant

Ppt 2-4
$Bi(OH)_3$(s) (white)

Soln 2-4
$Cu(NH_3)_4^{2+}$ (deep blue)

Add
sodium stannite reagent, $Sn(OH)_3^-$

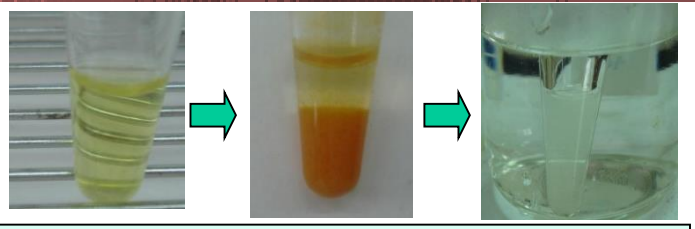
$2Bi(OH)_3(s) + 3Sn(OH)_3^- + 3OH^- \rightarrow \mathbf{2Bi}\downarrow + 3Sn(OH)_6^{2-}$

Bi(s) (black)

✓ **Prepare sodium stannite freshly in one test tube:**

$SnCl_2(aq) + NaOH(aq) \rightarrow Sn(OH)_2(s) \xrightarrow{\text{Excess NaOH}} Sn(OH)^-_3(aq)$

Step 4/4: Identifying Sn^{4+} and Sb^{3+}



Soln 2-2 Arsenic subgroup
SbS_3^{3-}, SbO_3^{3-}, SnS_3^{2-}, SnS_2OH^-

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

$SbS_3^{3-} + H^+ \rightarrow Sb_2S_3\downarrow$
$Sb_2S_3\downarrow + H^+ + Cl^- \rightarrow 2\mathbf{SbCl_4^-}(aq)$

Soln 2-6
$SnCl_6^{2-}$, $SbCl_4^-$

Discard residue

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

$SnCl_6^{2-} + 3H_2C_2O_4 \rightarrow Sn(C_2O_4)_3^{2-}$ (stable)

$SbCl_4^- + H_2S \rightarrow Sb_2S_3$ (orange)

SbTest

¼ small spatula **$H_2C_2O_4$(s)**
2 d **13%TA**
Warm in water bath

Sb_2S(s) (orange)

Sn test

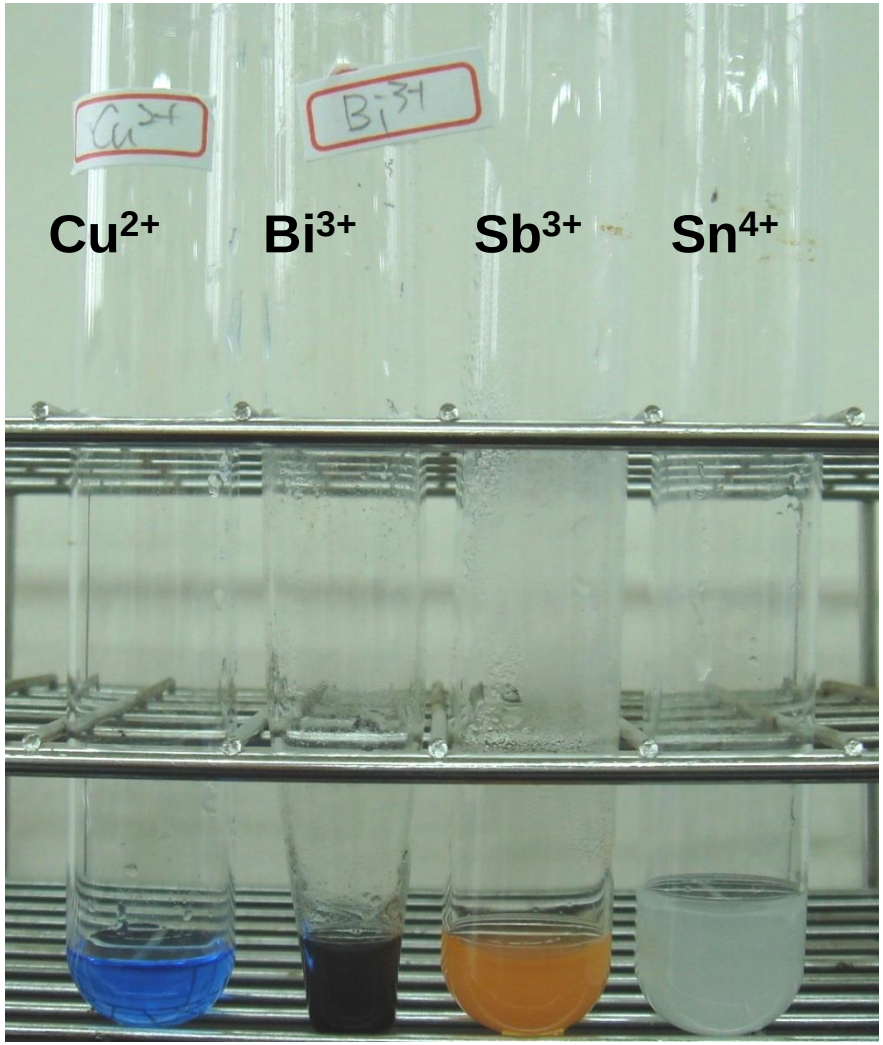
$Sn^{2+} + 2HgCl_2 \rightarrow \mathbf{Hg_2Cl_2}\downarrow + Sn^{4+} + 2Cl^-$

Small **Al** strip /6 M **HCl**
Heat in boiling water bath to dissolve
Cfg. and obtain soln
Add 0.1 M **$HgCl_2$** to solution

Hg_2Cl_2(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. NH_3(aq), and conc. HCl(aq) in the fume hood
- Take only the required amount of chemicals as lab manual to minimize chemical waste

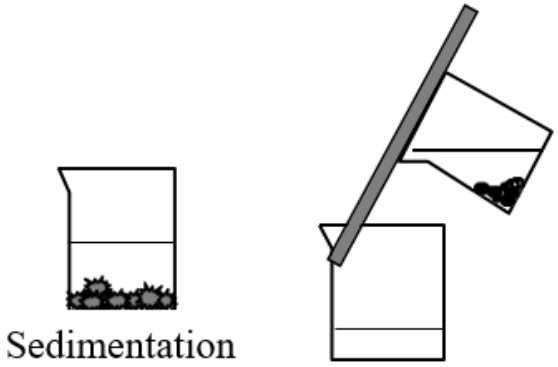
Clean-Up and Check-Out

- 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

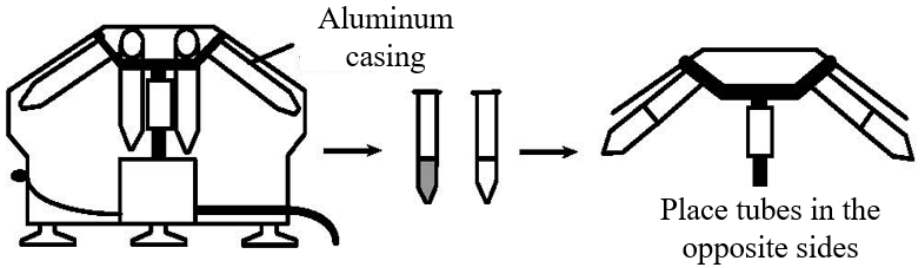
T5 - Decantation



- 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.

T5 Video (YouTube link)

T8 - Centrifugation

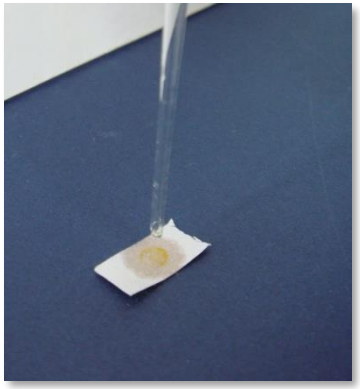


- Check the casing inside the machine is intact. If corrosion causes holes in casing or there is an unknown object inside, clean or replace the casing.
- Use centrifuge tubes in centrifugation, do not use test tubes.
- Centrifuge tubes should be placed in opposite sides to keep balancing.
- The lid should 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.

T8 Video (YouTube link)

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.



T15 Video (YouTube link)