Qualitative Analysis of Group I Cations

Collect:
• Centrifuge tubes (5)
• Test tube holder
• Dropper
• Latex gloves
• Labels

Prepare:
• Centrifuge (underneath lab bench)
• Test tube rack, test tubes, beakers
*Concentrated NH₃(aq): in hood
*Heating the water bath in hood
Objective and Techniques

- To learn the techniques of separating and identifying some common cations
- To understand the principles of precipitation and equilibrium of complex formation

Techniques

- Vortex mixer
- Precipitation
- Centrifuge
- Decantation
- Litmus and universal indicator paper

Vortex Mixer

Decantation
<table>
<thead>
<tr>
<th>Cationic Solutions</th>
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<tbody>
<tr>
<td>(I) Insoluble chlorides: Hg$_2^{2+}$, Ag$^+$, Pb$^{2+}$</td>
</tr>
<tr>
<td>(II) Insoluble sulfides in acidic medium: Hg$^{2+}$, Pb$^{2+}$, Cu$^{2+}$, Bi$^{3+}$, Cd$^{2+}$, As$^{3+}$, Sb$^{3+}$, Sn$^{4+}$</td>
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<tr>
<td>(III) Insoluble sulfide or hydroxides in alkaline medium: Al$^{3+}$, Fe$^{3+}$, Co$^{2+}$, Ni$^{2+}$, Cr$^{3+}$, Zn$^{2+}$, Mn$^{2+}$</td>
</tr>
<tr>
<td>(IV) Insoluble Carbonates: Mg$^{2+}$, Ca$^{2+}$, Sr$^{2+}$, Ba$^{2+}$</td>
</tr>
<tr>
<td>(V) Soluble cations: NH$_4^+$, Na$^+$, K$^+$</td>
</tr>
</tbody>
</table>
Cationic Solution

6 M HCl

Ppt of chlorides of group I cations
AgCl, Hg₂Cl₂, PbCl₂

Solution of group II~V cations

H₂S
pH 0.5

Ppt of sulfides of group 2 cations
HgS, CuS, Bi₂S₃, CdS, PbS,
As₂S₃, Sb₂S₃, SnS₂

Solution of group III~V cations

H₂S
pH 8

Ppt of sulfides or hydroxides of group III cations
Al(OH)₃, Fe(OH)₃, Cr(OH)₃, ZnS, NiS, CoS, MnS

Solution of group IV and V cations

(NH₄)₂CO₃
NH₃/NH₄Cl

Ppt of carbonates of group IV cations
MgCO₃, BaCO₃, SrCO₃, CaCO₃

Solution of group V cations
NH₄⁺, Na⁺, K⁺
**Procedure 1-1: Prepare Testing Solutions**

Take a centrifuge tube and labeled

Add $\text{Hg}_2^{2+}$, $\text{Ag}^+$, $\text{Pb}^{2+}$

(2, 2, 3 drops)

to prepare testing soln

* Methods for mixing solutions completely:
(1) Shake/flick, (2) Use Vortex mixer, (3) Use stirring rod
Procedure 1-1 Precipitation of Insoluble Chlorides

Cationic solution

Do not add too much HCl(aq), it may cause the chlorides to dissolve

step 1-1
(1) Add 2 drops 6 M HCl(aq), stir for 1~2 mins,
(2) Centrifuge and separate supernatant from ppt.

Note: Use Vortex mixer to help mixing thoroughly

Ppt 1-1
AgCl(s), Hg₂Cl₂(s), PbCl₂(s)
(white) (white) (white)
(1) Add 1 d. 6 M HCl with 10 d. of water to wash ppt,
(2) Centrifuge and separate ppt and supernatant

Soln 1-1
Add 1 drop 6 M HCl(aq)
Check whether precipitation is complete

No

Yes

Repeat steps 1-1
Discard in waste bin
**Procedure 1-2 & 1-3 Separation and Identification of Pb^{2+}**

**Step 1-2**
1. Add 5 d. distilled water and mix well
2. Heat in boiling water bath for several min. *in hood*
   (Water bath should bring to boil to extract PbCl\(_2\) efficiently)
3. Centrifuge to separate the ppt. and supernatant

**Ppt 1-1**
AgCl(s), Hg\(_2\)Cl\(_2\)(s), PbCl\(_2\)(s)

**Step 1-3-2**
Extract ppt 1-2 2~3 times and centrifuge to obtain the soln

**Ppt 1-2**
AgCl(s), Hg\(_2\)Cl\(_2\)(s)

**Soln 1-2-a**
Contains Pb\(^{2+}\)(aq)

**Step 1-3-1**
1 d. 6 M HOAc
1 d. 0.5 M K\(_2\)CrO\(_4\)

**PbCrO\(_4\)(s)**
(Yellow ppt)

**Ppt 1-1 should be extracted with hot water 2~3 times until adding K\(_2\)CrO\(_4\) to extracted soln slows slightly yellow ppt**

**Soln 1-2-b**
Contains Pb\(^{2+}\)(aq)

\[
Pb^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow PbCrO_4(s)
\]
Procedure 1-4 Separation and Identification of Hg$_2^{2+}$

**Step 1-4**

(1) Add 2~4 d. 15 M NH$_3$ (in the hood)
(2) Stir and mix well
(3) Centrifuge to separate ppt. and supernatant

**Ppt 1-2**

AgCl(s), Hg$_2$Cl$_2$(s)

**Ppt 1-3**

HgNH$_2$Cl(s), Hg$^0$
(White) (Black)

**Soln 1-3**

Ag(NH$_3$)$_2^+$(aq)

**Reaction Equations**

\[ \text{Hg}_2\text{Cl}_2(s) + 2\text{NH}_3(aq) \rightarrow \]
\[ \text{Hg} \downarrow + \text{HgNH}_2\text{Cl}(s) + \text{NH}_4^+(aq) + \text{Cl}^-(aq) \]

\[ \text{AgCl}(s) + 2\text{NH}_3(aq) \rightarrow \]
\[ \text{Ag(NH}_3)_2^+(aq) + \text{Cl}^-(aq) \]
Procedure 1-5: Separation and Identification of Ag⁺

Add 6 M HNO₃ until solution is acidic

Use stirring glass rod to drip on litmus paper

Ag(NH₃)₂⁺(aq) + Cl⁻(aq) + H⁺(aq) → AgCl(s) + 2NH₄⁺(aq)
Notice

- Prepare hot water bath on hot plate in the fume hood
- Wear latex gloves at all times
- Take the amount of chemicals according to lab manual, to reduce hazardous waste
- Use test tube rack or test tube holder to transport test tubes or centrifuge tubes
- It may produce hazardous acid/base fumes, **all the heating processes must be done in the fume hood**
- After centrifuging, solid precipitates and the supernatant should be separated by decantation
- **The liquid waste contains heavy metals and should be collected and discarded into the recycling bin**
Record detailed observations:

- Reaction conditions
- Operation (added drops, centrifuge speed, etc.)
- Phenomenon (color of precipitate and soln, reaction fast or slow, etc.)

* Keep results and show to TA
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 misfunction, 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.
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.