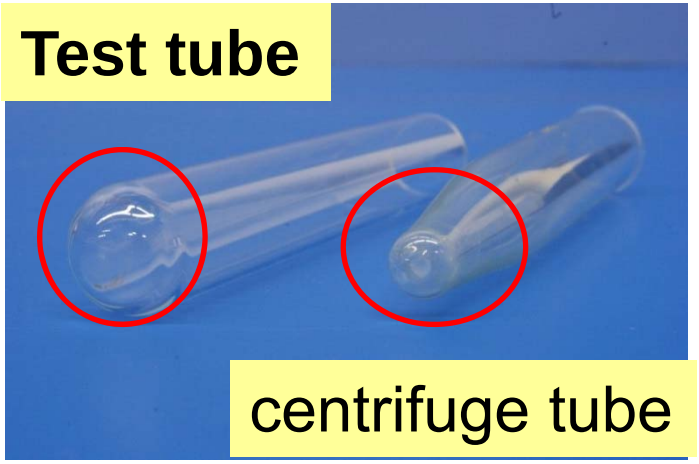
Qualitative Analysis of Group I Cations

(2015/09/28 revised)

Collect:

- Centrifuge tubes (5)
- Test tube holder
- Dropper
- Latex gloves
- Labels



Prepare:

- Centrifuge (underneath lab bench)
- Test tube rack, test tubes

***Concentrated NH_3(aq): in hood**

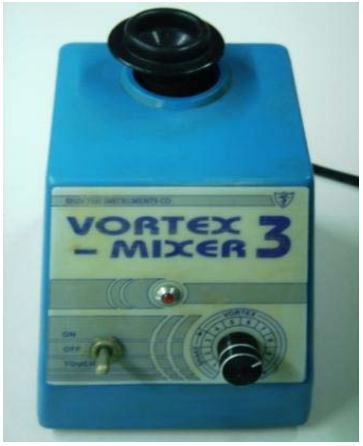
***Heating the water bath in hood**

Objective and Techniques

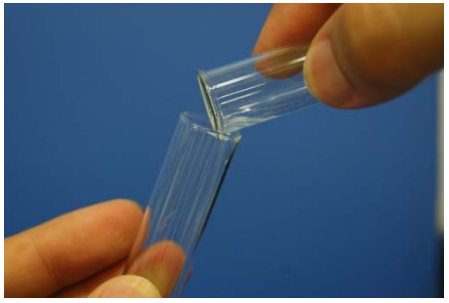
- To learn the techniques of separating and identifying some common cations
- To understand the principles of precipitation and equilibrium of complex formation

Techniques

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



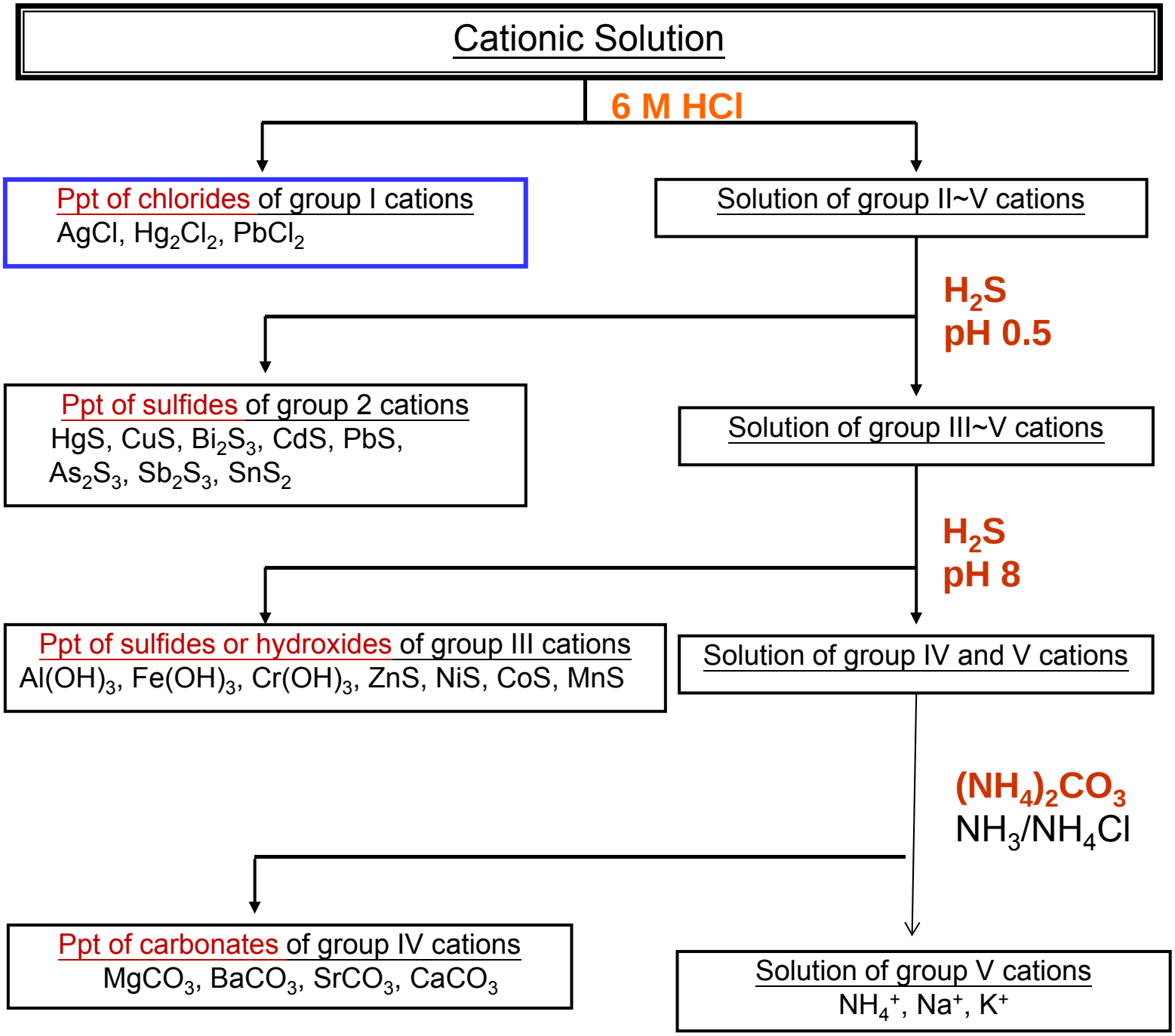
Vortex Mixer



Decantation

Introduction: Qualitative Analysis of Group I~V 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+}
(III)	Insoluble sulfide or hydroxides in alkaline medium: Al^{3+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cr^{3+}, Zn^{2+}, Mn^{2+}
(IV)	Insoluble Carbonates: Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}
(V)	Soluble cations: NH_4^+, Na^+, K^+

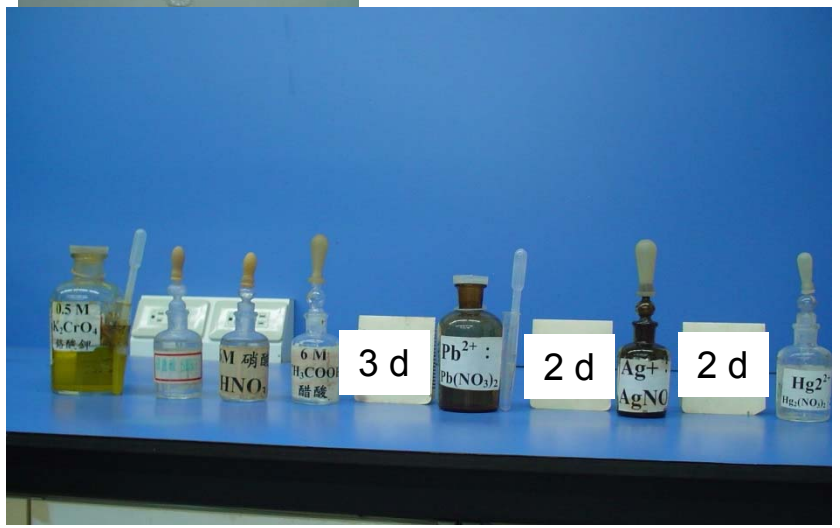

Cationic Solution
6 M HCl
Ppt of chlorides of group I cations
AgCl, Hg2Cl2, PbCl2
Solution of group II~V cations
H2S
pH 0.5
Ppt of sulfides of group 2 cations
HgS, CuS, Bi2S3, CdS, PbS,
As2S3, Sb2S3, SnS2
Solution of group III~V cations
H2S
pH 8
Ppt of sulfides or hydroxides of group III cations
Al(OH)3, 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 Hg_2^{2+}, Ag^+, Pb^{2+}
(2, 2, 3 drops)
to prepare testing soln



*** Methods for mixing solutions completely:**
(1) Use stirring rod, (2) shake/flick, (3) Use Vortex mixer

Procedure 1-1
Precipitation of Insoluble Chlorides

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

Cationic solution

step1-1

(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

$AgCl_{(s)}$, $Hg_2Cl_{2(s)}$, $PbCl_{2(s)}$
(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

Ppt 1-1

Soln (Discard)

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+}

Ppt 1-1 should be extracted with hot water 2~3 times and centrifuged until the extracted solution when added K_2CrO_4, solution is only slightly yellow

Ppt 1-1

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 1-2

$AgCl_{(s)}$, $Hg_2Cl_{2(s)}$

Step 1-3-2

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

Ppt 1-2

$AgCl_{(s)}$, $Hg_2Cl_{2(s)}$

Soln 1-2-a

Contains $Pb^{2+}_{(aq)}$

Soln 1-2-b

Contains $Pb^{2+}_{(aq)}$

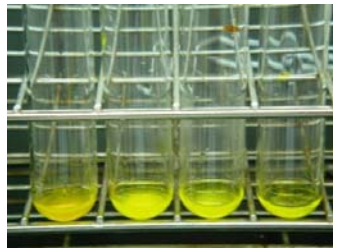
Step 1-3-1

Step 1-3-1

1 d. 6 M HOAc

1 d. 0.5 M K_2CrO_4

$PbCrO_{4(s)}$ (Yellow ppt)



$Pb^{2+}_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightarrow PbCrO_{4(s)}$

Procedure 1-4
Separation and Identification of Hg_2^{2+}

Ppt 1-2

$AgCl_{(s)}$, $Hg_2Cl_{2(s)}$

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 1-3

$HgNH_2Cl_{(s)}$, Hg^0

(White) (Black)

Soln 1-3

$Ag(NH_3)_2^+{}_{(aq)}$

$Hg_2Cl_{2(s)} + 2NH_{3(aq)} \rightarrow$

$Hg\downarrow + HgNH_2Cl_{(s)} + NH_4^+{}_{(aq)} + Cl^-{}_{(aq)}$

$AgCl_{(s)} + 2NH_{3(aq)} \rightarrow$

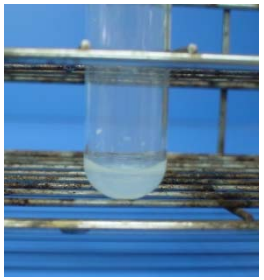
$Ag(NH_3)_2^+{}_{(aq)} + Cl^-{}_{(aq)}$

Procedure 1-5: Separation and Identification of Ag^+

Soln 1-3

$Ag(NH_3)_2^+{}_{(aq)}$

Add 6 M HNO_3
until solution is acidic



$AgCl_{(s)}$
(White Precipitate)

Use stirring glass rod to drip on litmus paper

$$Ag(NH_3)_2^+{}_{(aq)} + Cl^-{}_{(aq)} + H^+{}_{(aq)} \rightarrow AgCl_{(s)} + 2NH_4^+{}_{(aq)}$$

Notice

- Prepare hot water bath on hot plate in the hood
- Wear latex gloves throughout experiment
- Take the amount of chemicals according to lab manual, to avoid pollution
- Use test tube rack or test tube holder to transport test tubes or centrifuge tubes; do not use your bare hands
- The experiment produces toxic fumes, **all the heating processes must be done under the 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**

Manipulate the Centrifuge

- Use centrifuge tubes in centrifuge, **do not use test tubes**
- Centrifuge tubes should be placed in **opposite sides to keep balanced**
- The lid should be closed during use; the centrifuge should be started from **slow** to check if there are unusual sounds, then the speed can be increased
- If there are unusual sounds or movement in the centrifuge, the power should be turned off first to fix
- There must be at least one person watching the centrifuge when in use
- Centrifuge for approx. **1~2 min**, open lid when the centrifuge has completely stopped