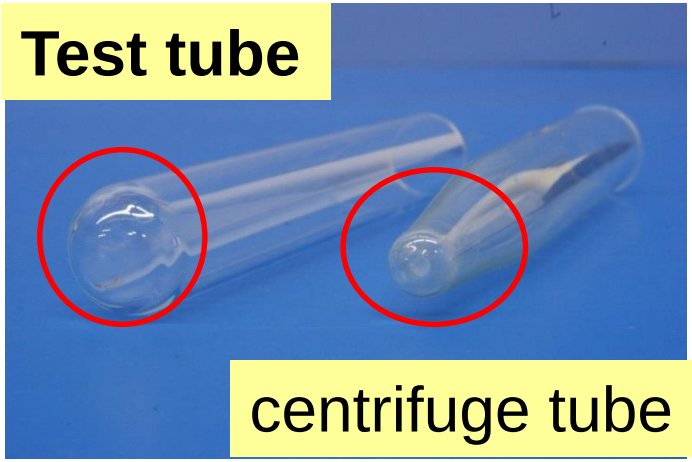
Qualitative Analysis of Group I Cations

(2021/03/02 revised)

Collect:

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



Prepare:

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

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

***Heating the water bath in hood**

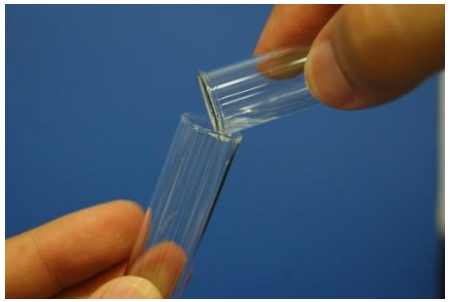
Objective and Techniques

- 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
- Centrifugation
- Decantation
- Litmus and universal indicator paper

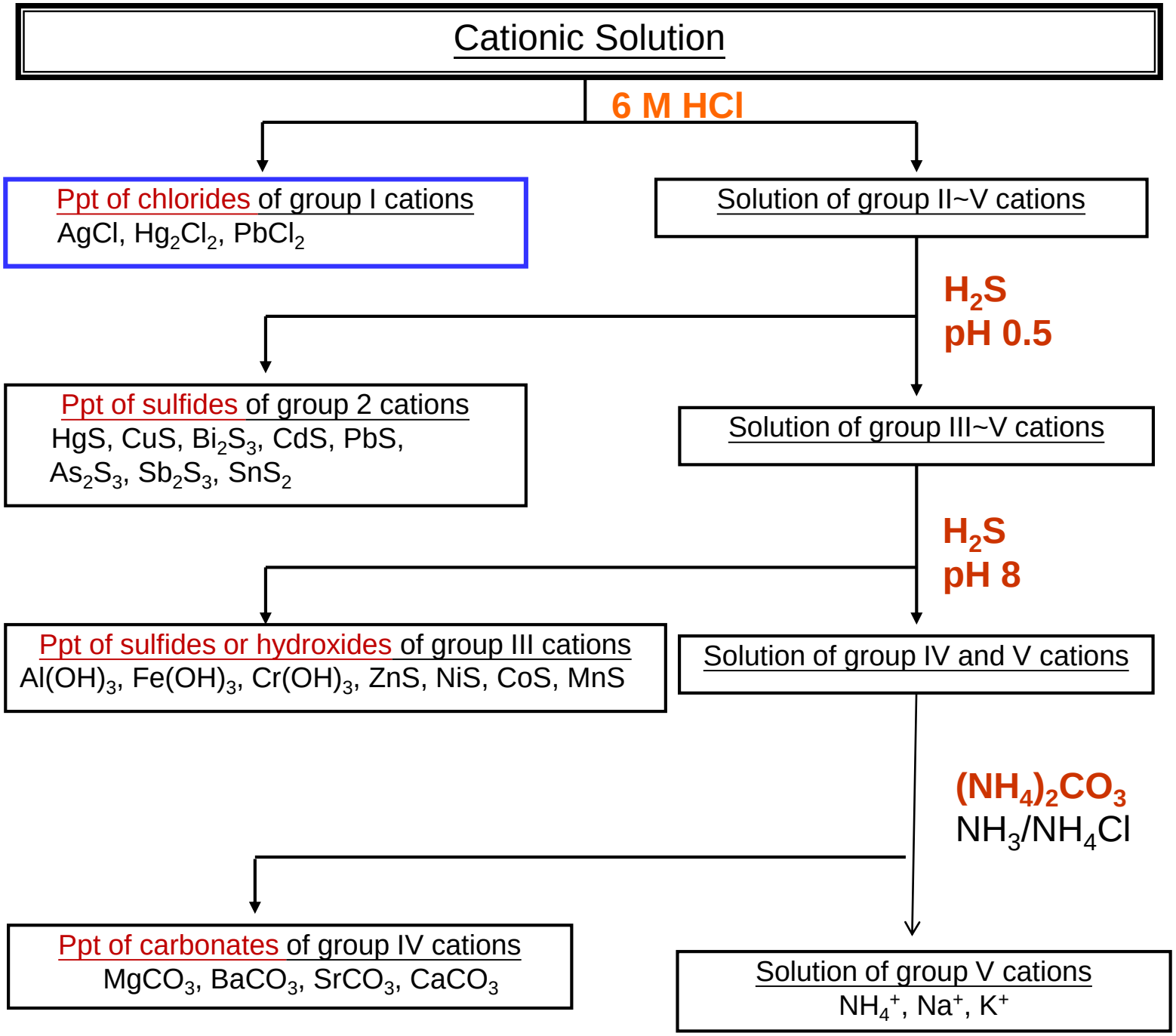
Vortex Mixer



Decantation

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^+

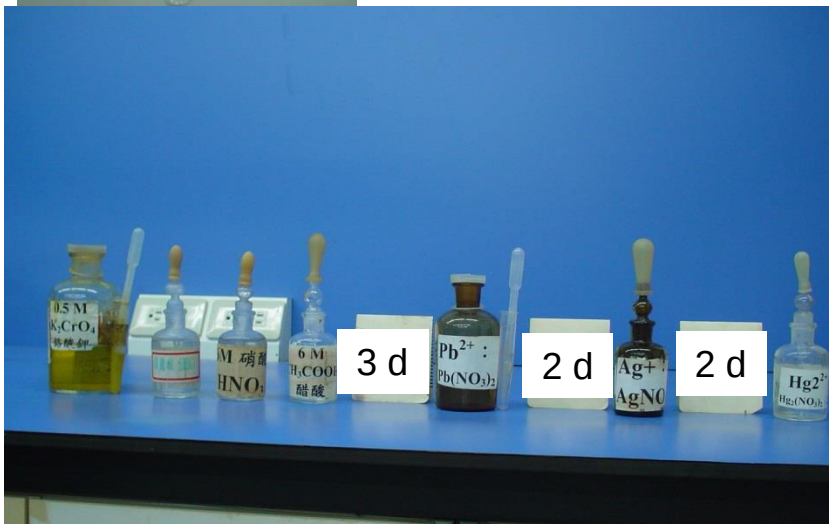

Cationic Solution
6 M HCl
Ppt of chlorides of group I cations
AgCl, Hg_2Cl_2, $PbCl_2$
Solution of group II~V cations
H_2S
pH 0.5
Ppt of sulfides of group 2 cations
HgS, CuS, Bi_2S_3, CdS, PbS,
As_2S_3, Sb_2S_3, SnS_2
Solution of group III~V cations
H_2S
pH 8
Ppt of sulfides or hydroxides of group III cations
$Al(OH)_3$, $Fe(OH)_3$, $Cr(OH)_3$, ZnS, NiS, CoS, MnS
Solution of group IV and V cations
$(NH_4)_2CO_3$
NH_3/NH_4Cl
Ppt of carbonates of group IV cations
$MgCO_3$, $BaCO_3$, $SrCO_3$, $CaCO_3$
Solution of group V cations
NH_4^+, 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) Shake/flick, (2) Use Vortex mixer, (3) Use stirring rod

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
$AgCl(s)$, $Hg_2Cl_2(s)$, $PbCl_2(s)$

Soln

Soln 1-1

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

No → **Repeat steps 1-1**

Yes → **Keep in tube**

Procedure 1-2 & 1-3 Separation and Identification of Pb^{2+}

Ppt 1-1
$AgCl(s)$, $Hg_2Cl_2(s)$, $PbCl_2(s)$

Ppt 1-1 should be extracted with hot water 2~3 times until adding K_2CrO_4 to extracted soln shows slightly yellow ppt

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
$AgCl(s)$, $Hg_2Cl_2(s)$

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

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

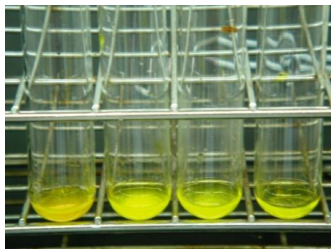
Step 1-3-1
1 d. 6 M HOAc
1 d. 0.5 M K_2CrO_4

Ppt 1-2
$AgCl(s)$, $Hg_2Cl_2(s)$

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

Step 1-3-1

$PbCrO_4(s)$ (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

$Ag(NH_3)_2^+(aq)$

$Hg_2Cl_2(s) + 2NH_3(aq) \rightarrow$

$Hg\downarrow + HgNH_2Cl(s) + NH_4^+(aq) + Cl^-(aq)$

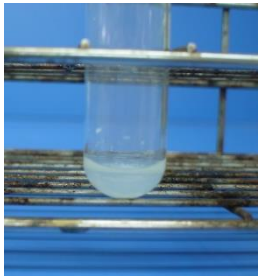
$AgCl(s) + 2NH_3(aq) \rightarrow$

$Ag(NH_3)_2^+(aq) + Cl^-(aq)$

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

Soln 1-3
$Ag(NH_3)_2^+(aq)$

Step 1-5
Add 6 M HNO_3
until solution is acidic



AgCl(s)
(White Precipitate)

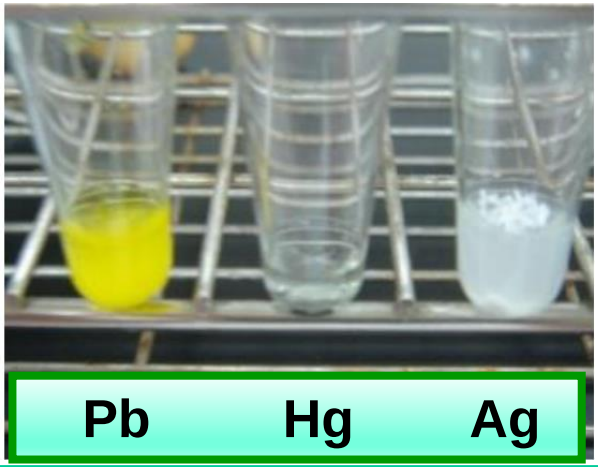
Use stirring glass rod to drip on litmus paper

$$Ag(NH_3)_2^+(aq) + Cl^-(aq) + 2H^+(aq) \rightarrow AgCl(s) + 2NH_4^+(aq)$$

Keep Results for Inspection

Record detailed observations:

- Operation (i.e. added drops, centrifuge speed, repeat extraction three times, etc.)
- Reaction conditions (i.e. in fume hood, in boiling water bath, etc.)
- Phenomenon (i.e. color of precipitate and slon, respectively, reaction fast or slow, etc.)



*** Keep results and show to TA**

Notice

Condensed Report

- Prepare hot water bath on hot plate in the fume hood
- Wear latex gloves throughout experiment
- Take the amount of chemicals according to lab manual, to reduce hazardous pollution
- Use test tube rack or test tube holder to transport test tubes or centrifuge tubes
- It may produces 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**
- **Remove the labels and wash tubes before returning**
- **Clean up the lab bench and tuck lab stools in**

POST-LAB Assignments

- When accomplish the experiment:
  1) FIND **TA** to check your lab record and get signature (Hand in lab certification and lab report)
  2) Find **Assistant TA** to check your apparatus and clean up, then get signatures
  3) **GROUPS ON DUTY** shall help TA to recover the lab environments
- Take off lab coat and goggles only when you STEP OUT LAB
- **Lab report (condensed report):**
  - **Subgroup A (person in charge): summit lab report including prelab report, lab records, and results**
  - Subgroup B: summit your prelab report

Scoring Rubrics for Lab Report

Brief report

Category	Guidelines
I. Prelab report	1. Indicate the main principles and chemical equations in summary.
	2. List the chemicals' properties, appearance, and toxicity.
	3. Use the flow chart to explain the procedures concisely.
II. Lab record	4. Record the data with correct significant figures and units.
	5. Record the observations, operation, and reaction condition in details.
III. Results & discussion	6. Process the data correctly, including the equations and calculation.
	7. Indicate the final results with correct significant figures and units.
	8. Analyze the results and make the appropriate discussion.
	9. Plot the results with appropriate XY axis and indicate each curves.
	10. Provide the concrete outcome and constructive suggestion.

Vortex Mixer

Notes:

1. **Before use:**
   Switch to “TOUCH” mode and set speed knob at low
2. **During use:**
   Bring tube bottom to touch the tube holder on mixer. Adjust to moderate speed during oscillation because high speed may let the mixed solution splash out

Use moderate speed during oscillation

Tube holder

“ON”

“TOUCH”

Mode switch

Speed knob

T8 - Centrifugation

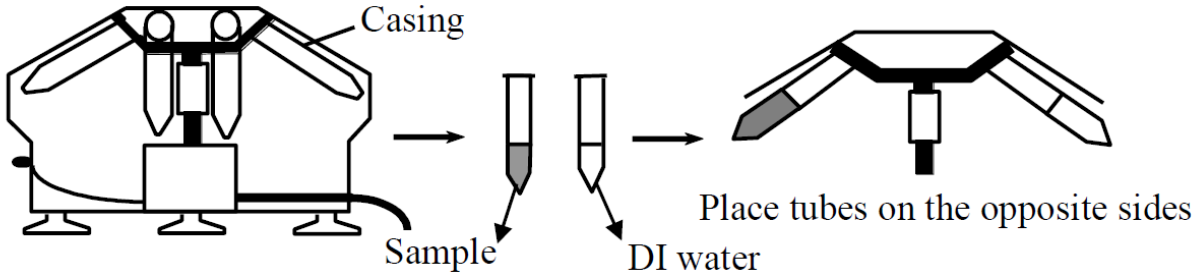


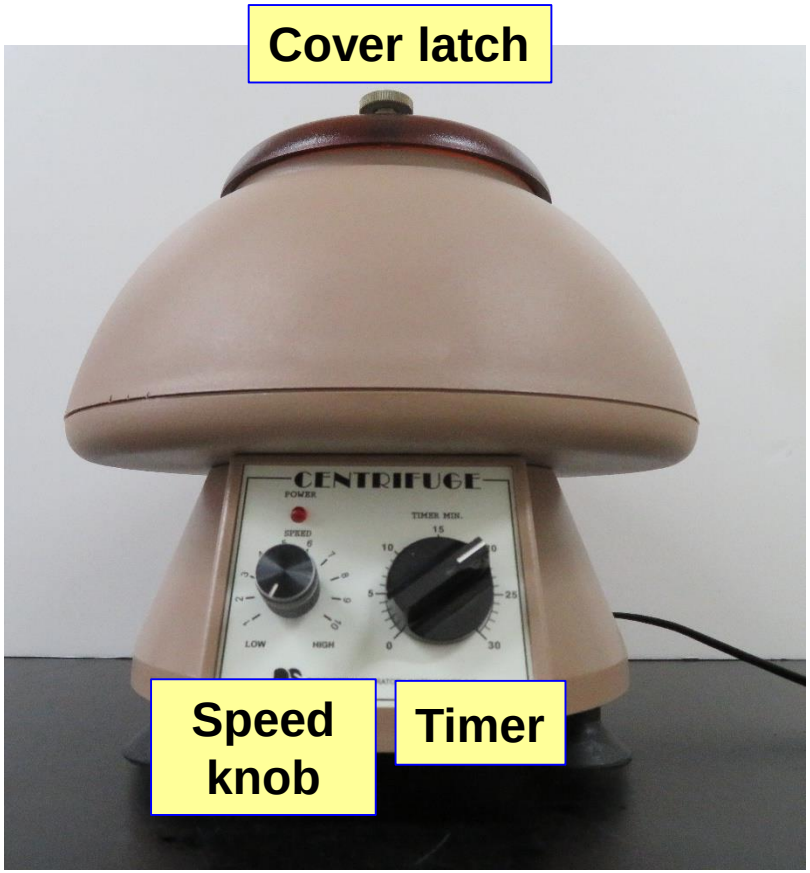
Figure T8-1 Section view of a centrifuge

T8 Video on YouTube (Click)

- Check the casing inside the machine is intact and clean.
- Use centrifuge tubes in centrifugation; **do not use ordinary test tubes**.
- Use an equal number of tubes or fill one with a counterbalancing solution. Place centrifuge tubes on **opposite sides to keep balancing**.
- Always close the centrifuge cover before you start the motor, and open it only after the assembly has stopped.
- Start the centrifuge from low speed to check if there is any malfunction, then speed it up.
- If there are unusual sounds or vibration, turn off the centrifuge immediately in order to check and fix up.
- There must be at least one person look after the centrifuge when in use.
- When centrifugation is completed, turn off the switch and allow the rotating centrifuge assembly to come to rest. Do not attempt to stop the rotation manually when the centrifuge is still rotating at high speed.

Table Top Centrifuge

1. **Before centrifugation:**
   Place centrifuge tubes in equilibrium position.
   Latch the top cover, set speed knob at low scale **(1~2)**
2. **Start centrifugation:**
   Set timer at a short interval (1~2 min) to start centrifugation. If there are unusual sounds or vibration, stop the centrifuge immediately.
3. **Speed up:**
   Without any malfunction, then speed it up. **(scale 5)**
4. **Stop centrifugation:**
   Unplug the power cord or wait until time is up, never turn the timer counterclockwise to stop and cause malfunction



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T5 - Decantation

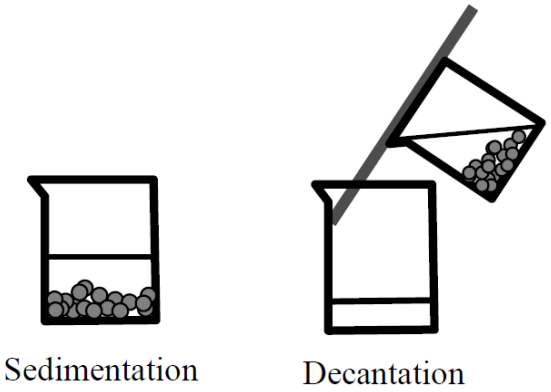
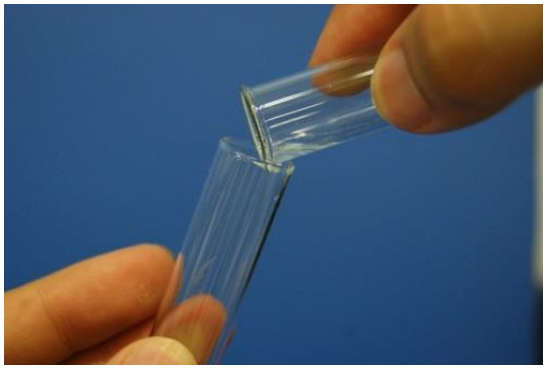


Figure T5-1 Sedimentation and decantation



T5 Video on YouTube (Click)

- Decantation is a simple method to separate solids and liquid.
- When specific gravity of the solid precipitate is greater than liquid, it settles to the bottom.
- While there is little solid remain suspended, it may be separated easily from the liquid by carefully pouring off the liquid.
- Stand the suspended solution by allowing the solid to settle to the bottom of the mixture.
- Use a glass rod to guide the liquid flow when pour off the liquid from the beaker slowly enough that the solid is not carried along.

T15 - Litmus Paper

T15 Video on YouTube (Click)

Figure T15-1 Using litmus paper to test acid base property

- Blue litmus paper will turn red under acidic condition.
- Red litmus paper will turn blue under basic condition.
- With the color change of the litmus paper, the acidity and alkalinity of sample can be known but the precise pH value cannot be determined.
- There is another widely used universal indicator paper which is a combination of a variety of indicators to obtain various color changes. Compare the colors with pH paper indicator chart, the pH value of sample can be known roughly.
- When test with solution, use a clean glass rod dip the solution and then touch it on a litmus paper or a universal indicator paper. Do not throw litmus paper directly into solution to avoid contamination.
- When test with gas, first wet the litmus paper and then place it on the opening of vessel. After the gas diffuse out and absorbed by litmus paper, the acidity and alkalinity can be judged by color change.

Chlorides of Group I Cations: Solubility vs Temperature

Temp. (°C) / Chlorides	Solubility (g/100 g H_2O)				
	0	10	20	50	100
$PbCl_2$	0.67	0.80	0.97	1.64	3.23
AgCl	$0.7*10^{-3}$	$1.1*10^{-3}$	$1.6*10^{-3}$	$5.4*10^{-3}$	$21*10^{-3}$
Hg_2Cl_2	$1.4*10^{-3}$	$1.7*10^{-3}$	$2.4*10^{-3}$		

Ref: 林洪志，分析化學，94頁，三民書局