Qualitative Analysis of Group 1 Cations

Last revised: 2024/10/16

Preparation

- Put on your lab coat and safety goggles
- Turn off your mobile phone
- Place your backpack in the drawer or the cabinet
- Put your prelab on the lab bench (hold it down with something heavy) for ATA to sign

Collect the following items

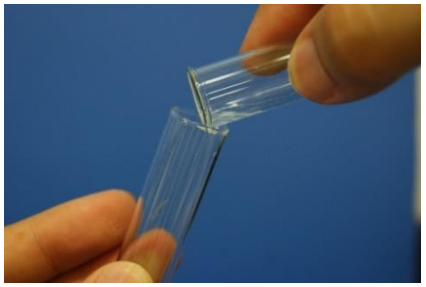
- ❑ Five centrifuge tubes, test tube tong
- ❑ Dropping pipets
- ❑ Sticky labels
- ❑ Centrifuge (under lab bench)

Objective and Principles

- **Objective:** Separate and identify common Group 1 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



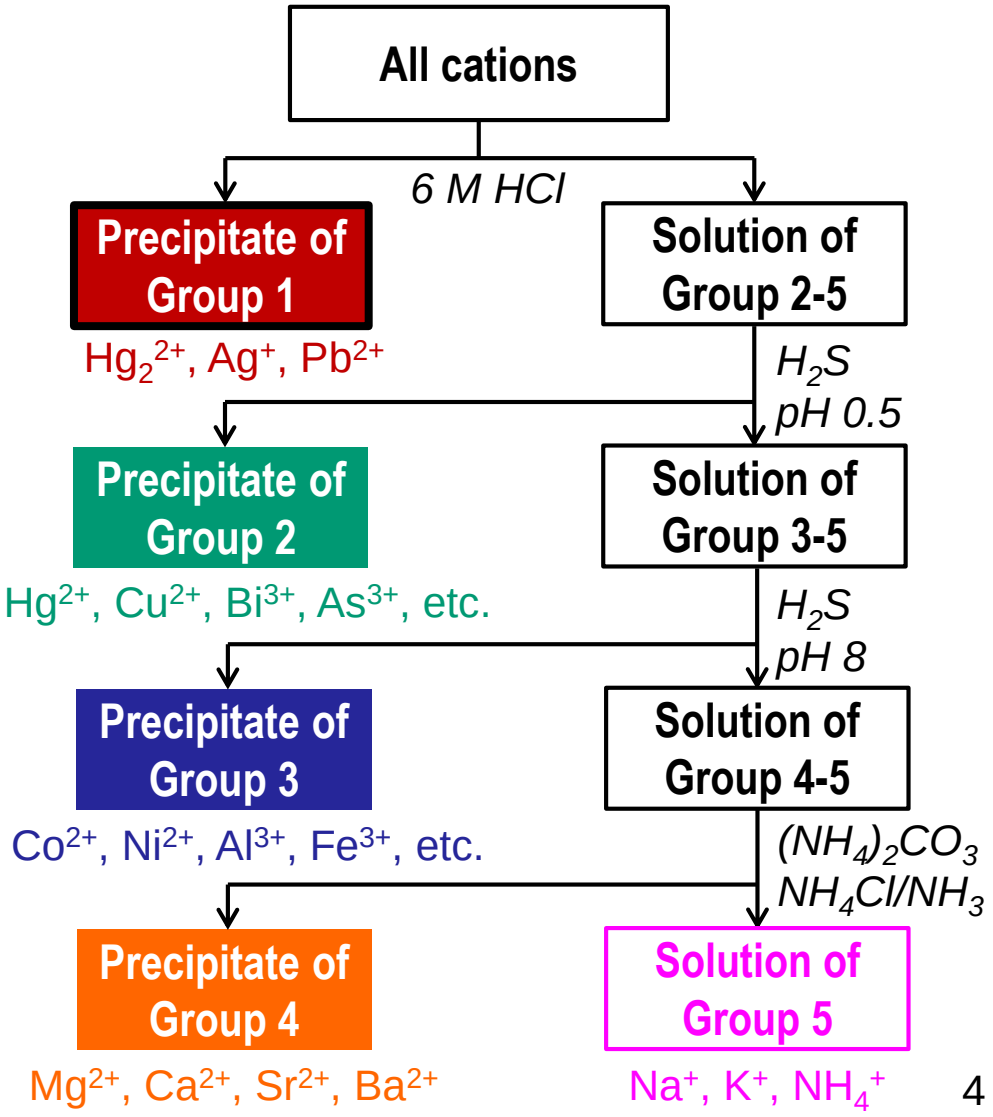
Decantation

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 Group 1 Cations

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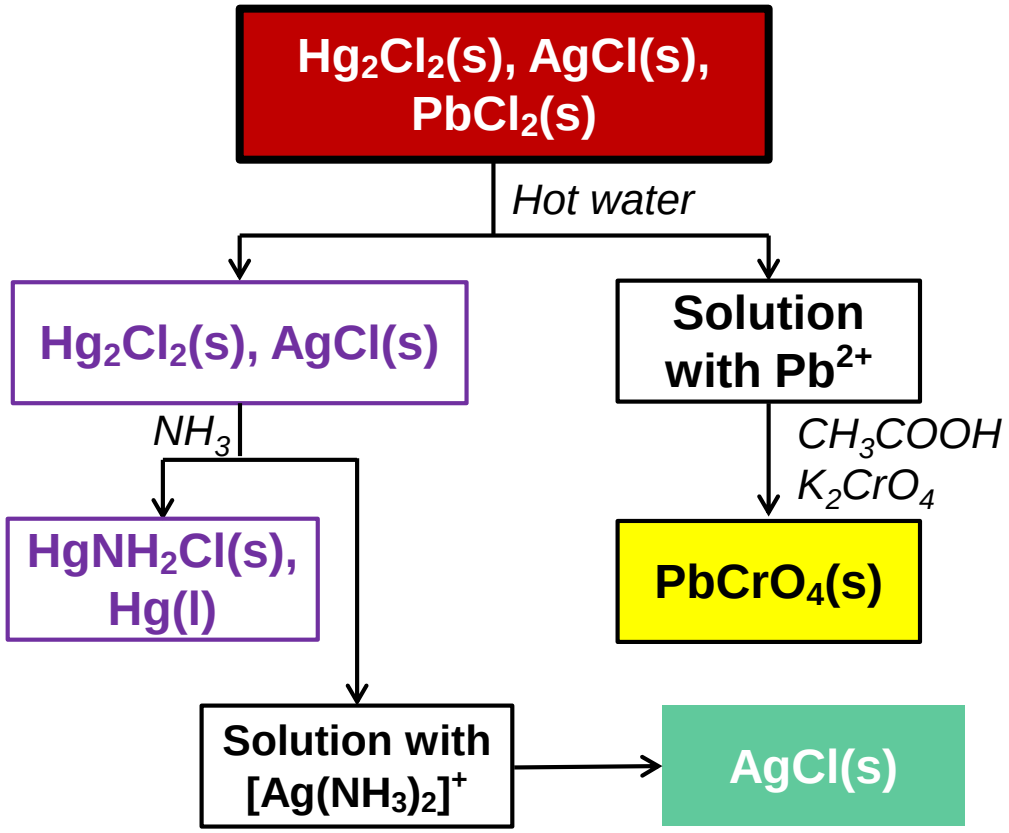


Step 1/4: Precipitaing Chlorides

- Label a centrifuge tube
- Mix 2 drops of Hg_2^{2+}, 2 drops of Ag^+, and 3 drops of Pb^{2+}
- Add 2 drops of 6 *M* HCl(aq) and stir for 1-2 min (finger-flicking, glass rod, or Vortex)
- Centrifuge and decant the supernatant

✓ Adding excess HCl(aq) may dissolve the chloride precipitates (why?)

✓ Clean glass rod thoroughly after each use

Step 1/4: Precipitation and Separation of Chlorides

Solution (Hg_2^{2+}, Ag^+, Pb^{2+})

- Add 2 drops of 6 *M* HCl(aq) and stir for 1-2 min
- Centrifuge and decant the supernatant into another centrifuge tube

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

- Wash with 1 d. 6 *M* HCl(aq) + 10 d. H_2O
- Centrifuge and decant the supernatant

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

Soln

Soln 1-1

- Add 1 d. 6 *M* HCl(aq)
- Precipitation is complete?

No

Yes

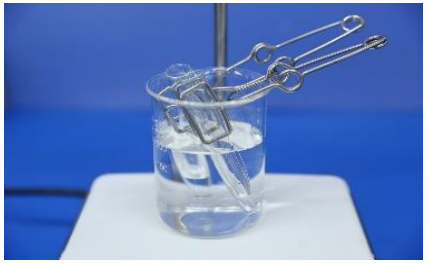
Keep in tube

Solubility of Chlorides

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	$1.4*10^{-3}$	$1.7*10^{-3}$	$2.4*10^{-3}$		

Step 2/4: Separation and Identification of Pb^{2+}

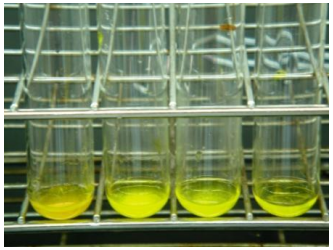
Ppt 1-1
$Hg_2Cl_2(s)$, $AgCl(s)$, $PbCl_2(s)$



- Add 5 drops DI water
- Heat in boiling water bath for 3 minutes in fume hood
- Centrifuge as quickly as possible (decant to a test tube)
- Repeat hot water extraction 2-3 times (supernatant should be free of Pb^{2+})

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

Soln 1-2



Add 1 d. 6 *M* CH_3COOH & 1 d. 0.5 *M* K_2CrO_4 to each test tube

$PbCrO_4(s)$ (yellow ppt)

$$Pb^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow PbCrO_4(s)$$

Step 3/4: Isolating and Identifying Hg_2^{2+}

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

- Add 2-4 d. conc. $NH_3(aq)$ in fume hood
- Stir with a glass rod
- Centrifuge to separate the supernatant

Ppt 1-3
$HgNH_2Cl(s)$, $Hg(l)$

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

$$Hg_2Cl_2(s) + 2\ NH_3(aq) \rightarrow Hg(l) + HgNH_2Cl(s) + NH_4^+(aq) + Cl^-(aq)$$

$$AgCl(s) + 2\ NH_3(aq) \rightleftharpoons [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$$

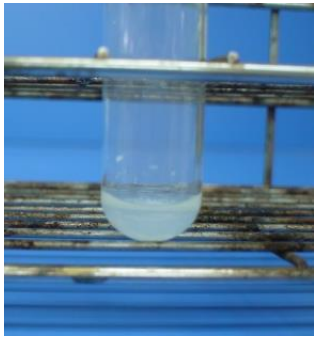
Step 4/4: Isolating and Identifying Ag^+

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

↓

Gradually add 6 *M* HNO_3 until the solution turns acidic (use a glass rod to spot the solution on a litmus paper)

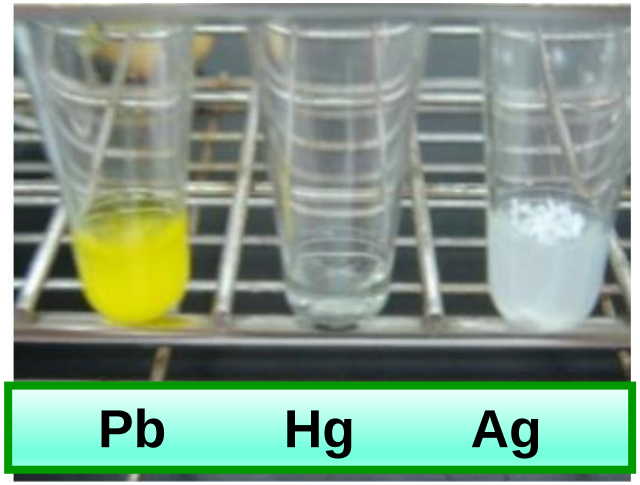
AgCl(s)
(white ppt)



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

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



Record Detailed Observations

Result \ Step		1-1	1-2		1-3	1-4
		6 *M* HCl	Boiling water bath	K_2CrO_4(aq) HOAc(aq)	Conc. NH_3(aq)	6 *M* HNO_3
Precipitate	Color					
	Chemical formula					
Supernatant	Color					
	Chemical formula					

Conclusion: Use 1-2 sentences to summarize your experiment formally

Safety Notes

- Wear NBR gloves throughout the experiment
- Do not attempt to decelerate the centrifuge by yourself – patience is a virtue
- Hot water bath and conc. $NH_3(aq)$ should be operated in the fume hood
- Take only the required amount of chemicals as lab manual (minimize chemical waste)
- Use test tube rack (or a test tube tong) for transporting test tubes and centrifuge tubes
- All wastes should be disposed into the heavy metal recycling container

Clean-Up and Check-Out

- Clean and return the centrifuge tubes (sticky labels should be removed)
- Tuck the lab stools underneath the lab bench
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- Present all identification products to TA during check-out

- This is a **Brief Report** experiment:
  - Give the experimental results and conclusion
  - Hand in the prelab/lab note/report together to the TA

- Groups on duty shall stay and help clean up the lab

T5 – Decantation

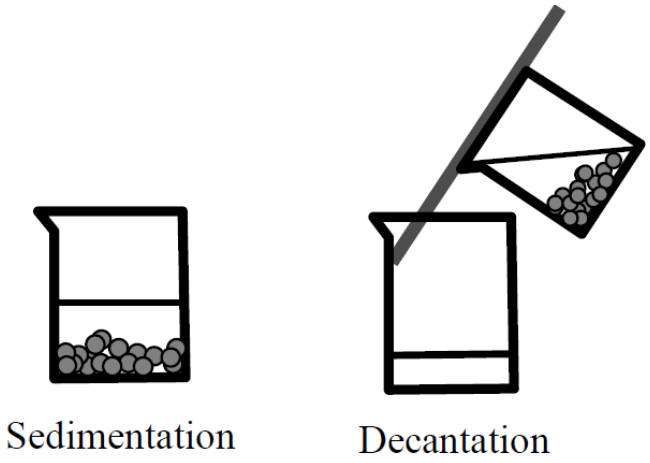
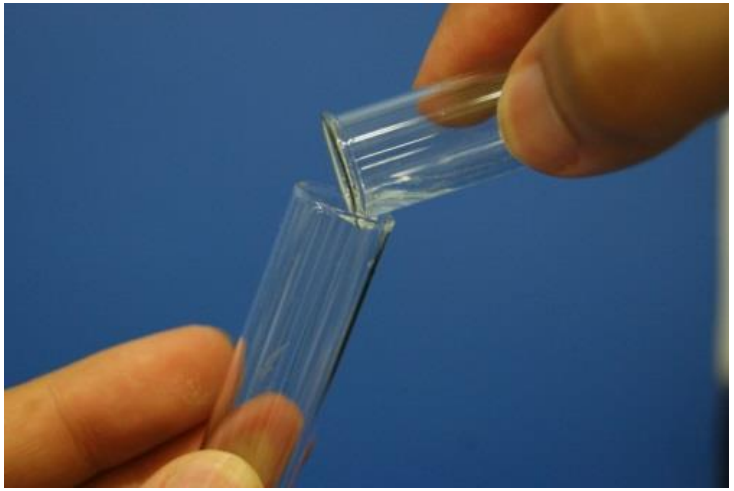


Figure T5-1 Sedimentation and decantation



Decantation between two centrifuge tubes

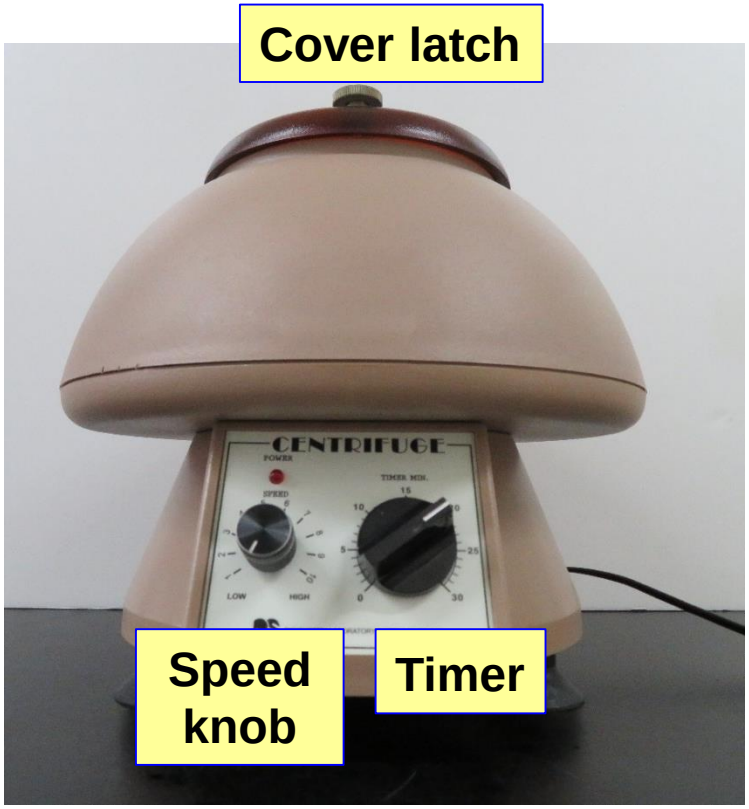
Decantation is a simple method for separting solid from liquid. The solid precipitate settles to the bottom if its specific gravity is greater than that of liquid. While there may still be some solid remaining suspended in the liquid, the separation can be achieved by carefully pouring the liquid off:

- Let the solid settle to the bottom of the mixture (or use a centrifuge – see T8)
- Pour the liquid out of beaker and use a glass rod to guide the liquid flow (Figure T5-1). This must be done slowly so that no solid is carried over.

T5 Video (YouTube link)

Table-Top Centrifuge

1) Before centrifugation: Arrange centrifuge tubes in a balance configuration. Close the cover latch, and set the speed to 1-2
2) Start centrifugation: Set the timer to 1-2 min to start the motor. In cases of unusual sounds or vibrations occuring, stop the centrifuge immediately
3) Speeding up: Turn the speed knob up to 5
4) Stop centrifugation: Wait until the timer returns to zero, or unplug the power cord. (Do not rotate the timer knob counterclockwise -- this will cause malfunction!)



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T8 – Centrifugation

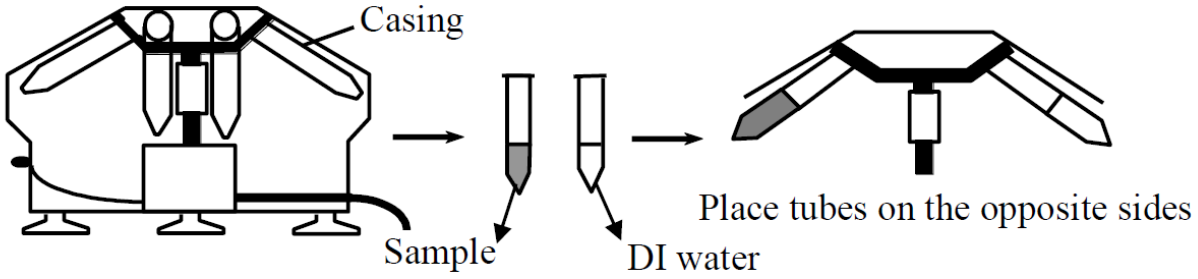

Figure T8-1 Section view of a centrifuge

T8 Video (YouTube link)

- Inspect whether the casings are still intact; clean or replace the casings if necessary
- Do NOT use regular test tubes in centrifuge; use only designated centrifuge tubes
- Mind the balance of the setup; only work with even number of tubes at the same time, and place them directly opposite to each other
- If only one sample solution needs to be centrifuged, take another tube with a similar amount of DI water and place it at the counterbalancing position
- Close the cover before starting the motor. Start the motor from low speed, then ramp up the speed if no malfunction is detected
- In cases of unusual sound or vibration occurring, turn the centrifuge off immediately
- Allow the rotating assembly to deaccelerate on its own after the process. Do NOT attempt to stop it manually, and open the cover only after the assembly has stopped

T15 – Litmus Paper

Blue → **Red** under acidic conditions

Red → **Blue** under basic conditions

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

- Color changes on litmus paper can only determine whether a solution is acidic or alkaline, but not the exact pH of the solution
- In addition to litmus paper, the *universal indicator paper* contains a variety of acid-base indicators and thus can exhibit more colors depending on the pH environment. Therefore the universal indicator paper can inform about the pH value better than the litmus paper
- Dip a clean glass rod into the solution to be tested, then use the same glass rod to touch the litmus paper or universal indicator paper (do not throw test papers directly into the solution). Observe any color change
- When testing gaseous species, wet a litmus paper then place it on the opening of the vessel containing the gas to be tested

T15 Video (YouTube link)