CH 223 Spring 2024: **"Qualitative Analysis of Group I Cations"** *(in class)* ***Lab:*** *Instructions*

Note: **This is the lab for section 01 and H1 of CH 223 only.**

- *If you are taking section W1 of CH 223*, please use this link:
  http://mhchem.org/q/8b.htm

Step One:

Get a printed copy of this lab! You will need a printed (hard copy) version of pages Ia-8-2 through Ia-8-7 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Bring the printed copy of the lab with you on Monday, May 20 (section 01) *or* **Wednesday, May 22 (section H1.)** During lab in room AC 2507, you will use these sheets (with the valuable instructions!) to gather data, all of which will be recorded in the printed pages below.

Step Three:

Complete the lab work and calculations on your own, then **turn it in** (page Ia-8-7 *only* to avoid a point penalty) **to the instructor on Wednesday, May 29 at 9 AM in AC 1303 (section 01)** *or* **Wednesday, May 29 at 1:10 PM in AC 2501 (section H1.)**

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Qualitative Analysis Of Group I Cations

In previous labs you have determined the amount of an unknown species present, such as percent chloride, identification of an unknown copper, and the molarity of various acid and base solutions. These experiments are a part of chemistry called *quantitative analysis*. When a chemist performs a **qualitative** analysis of a sample, s/he is more interested in the nature of the species present in a sample rather than the amount.

A set of experiments can be performed on an unknown mixture to precipitate cations in a sequential order. Under specific conditions, if a precipitate contains only one cation, the presence of that cation can be determined. Various types of reactions can be performed to separate the ions, including acid-base, complex ion formation, redox and other precipitation reactions. Ultimately, the sample should be resolved into fractions each containing one cation, whose presence is established by the formation of a characteristic precipitate or colored complex ion. The first step is to develop a scheme for the separation and identification of the cations.

Cations are typically divided into **Groups**, where each group shares a common reagent that can be used for selective precipitation. The classic qualitative analysis scheme used to separate various groups of cations is shown in the flow chart on the next page.

Over the next several weeks, you will study group I and III cations. You will develop a scheme to determine the cations present in an unknown mixture. Finally, you will carry out this scheme.

Precipitation and Separation of Group I Cations:

Pb^{+2}, Hg_2^{+2}, and **Ag^+** are all insoluble in cold water. They can be removed as a group from solution by the addition of HCl via simple precipitation in the following net ionic reaction:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

It is important to add enough HCl to ensure complete precipitation, but not too large an excess. In highly concentrated HCl solutions, chloro-complexes may form such as $AgCl_2^{-1}$.

Lead chloride can be separated from AgCl and Hg_2Cl_2 by heating with water, essentially reversing the above reaction. Once Pb^{+2} is in solution, you can discern its presence by adding chromate ion to produce a **yellow** solid:

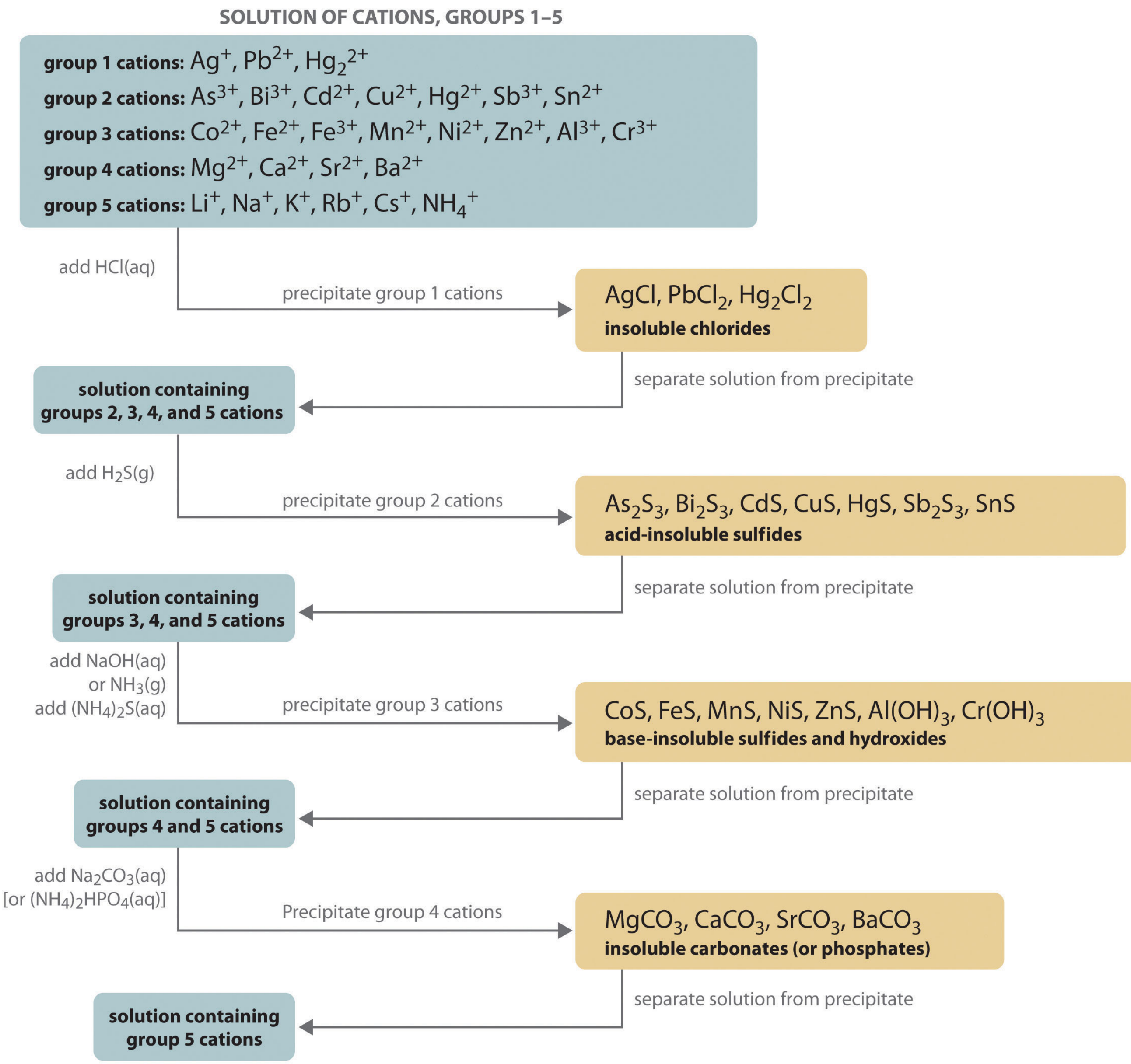
$$Pb^{+2}(aq) + CrO_4^{-2}(aq) \rightarrow PbCrO_4(\text{yellow solid})$$

Hg_2Cl_2 can be distinguished from AgCl by reaction with ammonia via oxidation reduction to yield finely divided black metallic mercury and a white complex compound $HgNH_2Cl$. As the reaction proceeds, the solid appears to change colors from white to **black** or **gray**:

$$Hg_2Cl_2(\text{white solid}) + 2\ NH_3(aq) \rightarrow NH_4^+(aq) + Cl^-(aq) + Hg(\text{black liquid}) + HgNH_2Cl(\text{white solid})$$

Silver chloride also reacts with aqueous ammonia to form a complex ion that remains in solution. Addition of a strong acid will destroy the complex and confirm the presence of silver ion by re-precipitating the **white** AgCl solid:

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



As you can see from the chart above, there are many Cation Groups.
We will analyze Group I Cations (the "Silver group") and some of the Group III Cations.

Laboratory Techniques: Above all else, **you *must* wear safety glasses at all times** while performing Qualitative Analysis labs! Failure to bring your own pair of safety glasses will result in a point penalty. Glasses must be worn over prescription glasses on these labs.

Attire in the Lab:
Please do not wear open toed shoes or sandals; shorts and shirts that show mid-riff areas are disallowed. You will be sent home to change if proper attire is not worn, so please, think safe when dressing for lab!

Waste:
Make sure all waste is placed in a proper waste container - do not pour anything down the drain.

Cleanliness:
Make sure that all test tubes, stirring rods, etc. are clean. Rinse all equipment with water before and after use. Use clean droppers.

Centrifuging:
Use centrifuge tubes when centrifuging - do not use ordinary test tubes. A tube of approximately the same mass and volume in the opposite slot of the centrifuge must be used to balance the centrifuge. Generally centrifuging for a minute or less is sufficient for these labs.

Decanting:
After centrifuging, the **supernatant** (the liquid above a precipitate; it is also called the *decantate*) is usually **decanted** (poured) into a clean test tube with the intent of keeping the solid in the original container. Carefully tip the test tube and pour off the supernatant without disturbing the solid. It may be poured directly, or a stirring rod may be placed across the mouth of the test tube to direct the supernatant into a clean test tube.

Washing a Precipitate:
After separation from the supernatant, a precipitate is often washed to free it from reagents that might interfere at a later stage. Usually, the rinse is deionized water, but other liquids or solutions may be used. After thorough stirring, centrifuge the sample and decant the wash solution.

Heating:
Due to the small quantity of material being heated, solutions in test tubes can reach its boiling point within a few seconds, and chemicals may be ejected violently from the container. If the solution volume is too large to fit in a centrifuge tube, reducing the volume can be accomplished by placing the solution in a 30 mL beaker and placing it on the hot plate. ***Watch the solution on the hot plate intently*** - if overheated, you may be left with a crusty remnant which is hard to reconstitute. Using a water bath is safer, but slower.

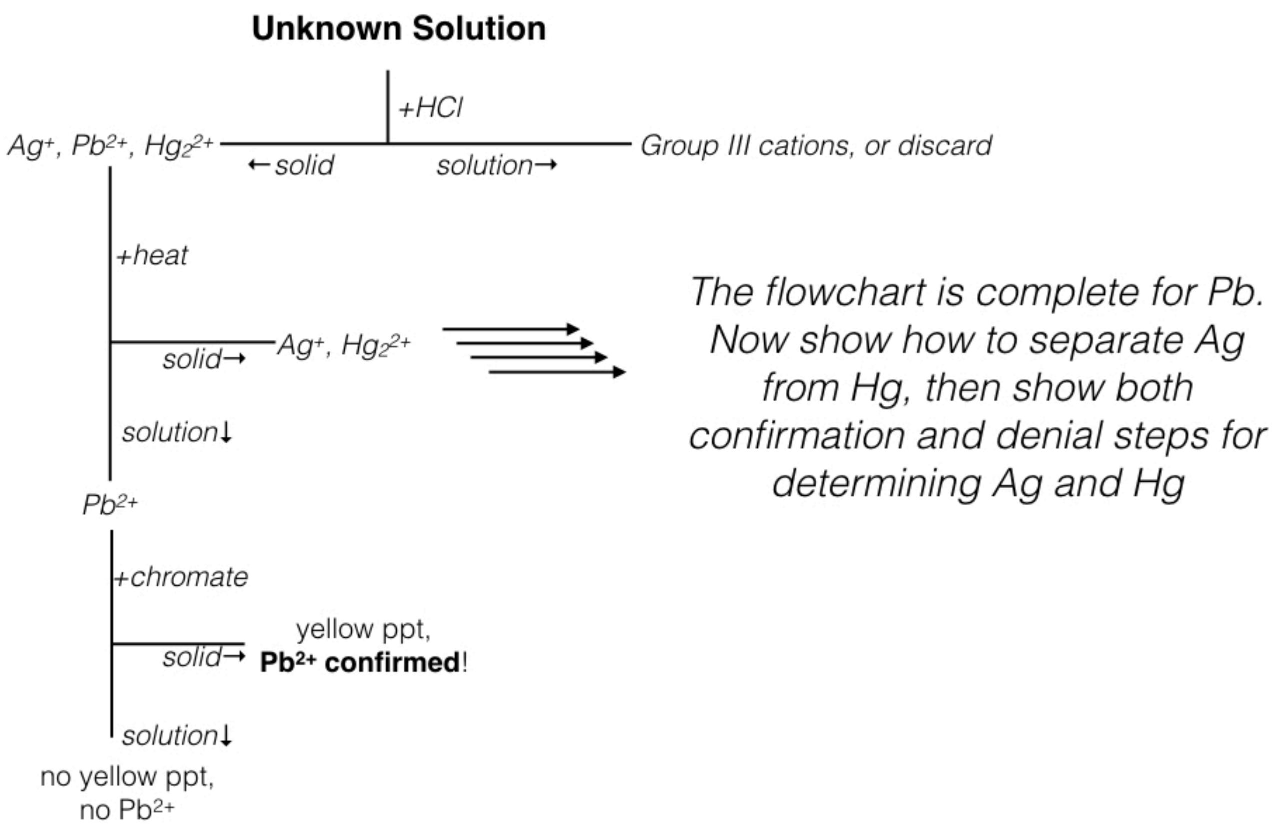
Testing pH:
When directed to check the pH of a solution, stir the solution thoroughly with a clean glass stirring rod and then touch the tip of the rod to a piece of litmus paper. Several such tests may be performed on each strip of paper. Red litmus paper will turn blue in basic solutions; blue litmus paper will turn red in acidic solutions.

Flow Charts:

It is possible to summarize the directions for the analysis of the Group I cations in a flow chart. In a flowchart, successive steps in the procedure are linked with arrows or lines. Reactant cations are at one end of the line; reagents and conditions used to carry out each step are placed alongside the lines. The line splits to show the two possible outcomes (yellow ppt = Pb^{+2} present; no ppt = absence). A flow chart for the separation of Group I cations should be included in your lab report.

A *partially* completed flowchart for the Group I Cations lab is included below. When you complete a flowchart, make sure that it shows clearly where each cation is separated out; also show the confirmation of each cation (with both yes and no options to indicate the presence or absence of the cation.)

Flowcharts may be completed on paper by hand or electronically on the computer; the choice is yours.



PROCEDURE: *All waste must go in a waste bottle. Safety glasses are mandatory for everyone; no open toed shoes or sandals, no shorts or mid-riff showing shirts.*

Step 1: Preparation:

Prepare a 1 mL "**known**" **sample** by placing 7 drops of each of the following into a centrifuge tube: 0.1 M $AgNO_3$, 0.3 M $Pb(NO_3)_2$ and 0.1 M $Hg_2(NO_3)_2$. Prepare a second 1 mL "**unknown**" **sample** using 1 mL of your unknown liquid. Be sure to write the identity of your unknown. Your unknown will have between one and three of the Group I cations in it, and the known will act as a "control" to see appropriate color changes, etc. throughout the tests.

You will perform the following tests upon *each* solution (both the known and unknown solutions) simultaneously. Be sure to write down any color changes, precipitates, etc. that you might observe in your lab notebook.

Step 2: Precipitation of Group I Cations:

Add two drops of 6 M HCl and mix with a clean stirring rod. Centrifuge the mixture being certain a centrifuge tube of equal volume is placed opposite your centrifuge tube as a balance. Add one more drop of 6 M HCl to the test tube to be certain of complete precipitation. Centrifuge again if necessary. Decant the supernatant into another test tube and save for later analysis for Group III cations (if appropriate, i.e. on the Final Lab). The precipitate should be white and contain the chlorides of the Group I cations.

Step 3: Separation and ID of Lead:

Rinse the precipitate from Step 2 with 1-2 mL of deionized water. Stir, centrifuge and discard the liquid. Add 2 mL deionized water and place in boiling water for two minutes, stirring occasionally to dissolve most of the $PbCl_2$. Centrifuge and decant the liquid into another test tube. Save the precipitate for later tests for silver and mercury.

Add one drop of 6 M acetic acid and two drops of 1 M K_2CrO_4 to the liquid. A bright yellow precipitate will appear if the lead (II) ion is present.

Step 4: Separation and ID of Mercury:

To the precipitate from step 3, add 1 mL 6 M NH_3 and stir. Centrifuge and decant the liquid into a test tube for testing for Ag^+ (Step 5.) A gray or black precipitate confirms the presence of the mercury(I) ion.

Step 5: Identification of Silver:

Add 6 M HNO_3 to the liquid from step 4 until it is acidic to litmus. Test for acidity by touching the end of your stir rod to litmus paper. If Ag^+ is present, it will precipitate in the acidified solution as white AgCl.

Step 6: Cleanup!

Please rinse all glassware and equipment prior to leaving lab. Return cleaned centrifuge tubes to the container without masking tape.

Include answers to the following postlab questions (most of which can be found on the introductory page for this lab, hint, hint ☺).

Qualitative Analysis of Group I Cations Lab - *Worksheet*

YOUR NAME: _______________________ ***LAB PARTNER(s):*** _________________

Unknown Number: ___________

Circle *either* yes or no for each metal cation in your unknown.

Pb^{2+}: yes no

Hg_2^{2+}: yes no

Ag^{+}: yes no

Include a flowchart showing the overall steps used to separate the Group I cations with your lab report.

Postlab Questions:

1. Write balanced net ionic equations for the following reactions:
   a. The precipitation reaction of the chloride of Hg_2^{+2} in step 2.

   b. The formation of the yellow precipitate in step 3.

   c. The formation of the black precipitate in step 4.

   d. The reaction that occurs in step 5.

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