

EXPERIMENT 11: Qualitative Analysis of Cations

Materials:

Equipment: centrifuge, test tubes (6 small, 2 medium), test tube rack, stirring rods, beral pipets, hot plate, small beaker, red litmus paper.

Reagents: 6M HCl, 6M NH₄OH, 1M KI, 6M CH₃COOH,
0.50 M known solutions of AgNO₃, Pb(NO₃)₂, and Hg₂(NO₃)₂
Unknown solutions, distilled hot water.

Objective:

Part I: Separate a mixture of known cations: Ag⁺, Pb²⁺ and Hg₂²⁺ into individual ions.

By performing a series of tests, become familiar with reactions characteristic for each cation, and their results.

Part II: Separate and identify individual cations in an unknown solution containing a mixture of two to three cations. Use information gathered in the **part I** to identify the cations.

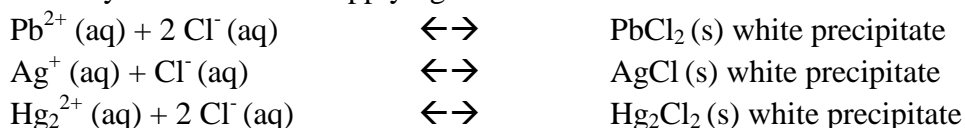
INTRODUCTION

The qualitative analysis is a general name for the methods used in the determination of the identity rather than the amount of chemical species (quantitative analysis). The qualitative process usually utilizes the reaction(s) characteristic for the given chemical species and interprets the obtained results using a deductive thought process. The qualitative analysis of cations requires an extensive knowledge of various aspects of chemistry, such as *acid-base equilibria*, *complex equilibria*, *solubility*, etc. However, in the deductive process, common sense and logic is as helpful as the knowledge of chemistry, if not more.

You will be working with a solution containing a mixture of cations. Before individual components of a mixture of cations can be successfully identified, they have to be separated from the mixture. The individual cations, once separated, may need to be identified in the ensuing *confirmatory reactions*. Using a flow chart and knowing the actual results of the reactions characteristic for a given cation (or a group of cations) makes it easier to identify this ion in the unknown sample, when the same results are obtained in a test. Therefore, it is useful to perform all of the characteristic reactions first on the mixture of ions whose identity is known, then proceed to the unknown sample.

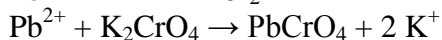
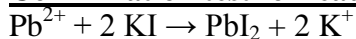
Reactions Characteristic for (Pb²⁺, Hg₂⁺², and Ag⁺)

Out the three cations in a mixture solution, all ions (Pb²⁺, Ag⁺, and Hg₂⁺²) will precipitate as PbCl₂, AgCl, and Hg₂Cl₂ when reacted with hydrochloric acid supplying chloride ion:

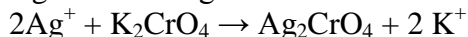
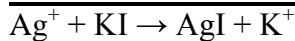


These formed solids are separated from the solution containing the other three ions by centrifuging the reaction mixture.

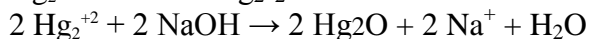
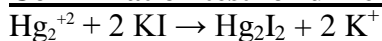
Confirmation test for lead:



Confirmation test for Silver:



Confirmation test for dimeric mercury ion:



General notes: Terms and techniques used in this experiment

1. Mixing solutions

After addition of any reagent to a solution, one must ensure proper mixing. To mix the reagent and the solution in a centrifuge tube, tap the bottom of the centrifuge tube against the table or snap it with your fingers while holding the upper part in the other hand.

2. Centrifuging

Precipitates are separated from the supernatant solution by centrifuging. This is the process of separating more dense solid particles from less dense liquid (solution) by spinning (separation by means of centrifugal force). The apparatus used here is called a centrifuge. It must be balanced to properly function. Balancing is done by putting the centrifuge tube containing the reaction mixture (tightly capped) in a sleeved centrifuge slot, then placing a centrifuge tube with an equal volume of tap water in a slot across from the first tube. Make sure that the tubes are tightly capped before they are placed in the centrifuge.

3. Decanting

The supernatant solution is transferred from above the precipitate to another tube by decanting. When a two-phase system (solid-liquid) is considered: after the solid settled to the bottom upon centrifuging, decanting is pouring the supernatant liquid out of the tube, leaving the solid behind.

4. Rinsing precipitates

All precipitates, after they have been separated from the supernatant solution, must be rinsed with distilled water before proceeding to the identification of the cation present. This process must be done to remove any cations present in the supernatant solution adhering to the solid. The presence of these ions may cause confusing results in the process of further identification or separation. The solid remaining after the supernatant solution has been removed is mixed with 10 drops of distilled water and the tube is tapped to thoroughly mix the contents. The tube is centrifuged, rinse water decanted, and the process repeated one to two more times.

It is VERY important that you take care to prevent contamination because of the large number of experiments that will be performed. Be sure to use deionized water in all chemical tests. Even the smallest amount of contamination can cause changes the results of your tests. This will lead you to record incorrect observations and to make wrong conclusions. As you are performing these experiments individually, you will have no one but yourself to blame for such errors. Following every test you should carefully clean the equipment with deionized water. Cleaning your test tubes and droppers following an experiment can prevent contamination on a subsequent experiment.

You should take care to label the vials which contain important stock solutions. Labels should be available in the laboratory. When testing how the cations behave to a specific reagent, perform tests on all ions simultaneously. Be sure that test tubes and/or the locations of the test tubes in the test tube rack are clearly labeled. Record observations immediately after each test. Following the test of all anions, check each test tube a second time to strengthen your observations.

When writing your observations, be sure to note the texture and color of precipitates and supernatant solutions. Are gases formed? Are reactions exothermic, endothermic, or is it difficult to tell? Remember, 'no reaction' is an important observation. Some combinations of reagents do not react. All observed reactions must include balanced chemical equations.

DIRECTIONS FOR CENTRIFUGE:

When using the centrifuge, do not use position #1. Be sure centrifuge is balanced with a second test tube with enough water, so that it weighs about the same as your sample test tube. Turn the timer to turn centrifuge on for one minute and wait for the timer to return to 0 to turn centrifuge off. Be sure centrifuge has stopped on its own before removing test tubes.

DO NOT POUR CHEMICALS DIRECTLY INTO THE CENTRIFUGE; THEY SHOULD BE IN SMALL TEST TUBES.

In this experiment you will determine what ions are present in a solution. To test for an ion, it must first be separated from other similar ions and then tested for. The separations are based on solubility differences. The ions we will be testing for are Ag^+ , Pb^{2+} , Hg_2^{2+} . This lab will consist of two parts. In Part I, you will follow the flow chart on the next page with ten drops of a known solution which contains all three ions. In Part II, you will be assigned an unknown solution which can contain one, two or all three ions. You will use ten drops of the unknown solution and follow the flow chart in the exact same way.

Report your observations and your conclusion about what ions are present in your unknown solution below.

PROCEDURE:

Part I: Known Solutions – Observation of Cations in Reagents

Obtain 1 mL (20 drops) samples of 0.5 M solutions of the salts **AgNO₃**, **Pb (NO₃)₂**, and **Hg₂ (NO₃)₂** as mixtures of **Ag⁺**, **Pb²⁺**, and **Hg₂²⁺** ions respectively in three separate clean, labeled vials or small test tubes. These will be your 'Test Solutions'. Identify the formulas and charges for the cations on your paper such as **Ag⁺**, **Pb²⁺**, and **Hg₂²⁺** ions respectively. To each test solution add 10 drops of reagent solutions such as **HCl**, **NH₄OH**, **KI**, and **CH₃COOH**. Each of the cation solutions should be subjected to each of the following tests. Record the cation and your observations following each experiment in space provided in steps 1-5 on report form.

Part II: Unknown Solutions - Identification of Cations in an Unknown Mixture

Obtain an unknown solution. The unknown solution contains between 1, 2, and 3 cations (or A,B, and C). After recording the **unknown number** _____ you are to analyze your unknown and identify the cations present. During the analysis you are to take careful notes of the procedures you follow, recording all tests and observations. After completing your analysis, explain which cations are present and which are absent and how you arrived at these conclusions. The unknown portion of the experiment will count heavily in determining your grade so take your time and be careful.

Take 20 drops of the unknown solution in a clean medium test tube and dilute the solution using 5 mL deionized water.

Add 10-15 drops of 6.0 M HCl. If there is no precipitate, go to next step in the provided flow chart.

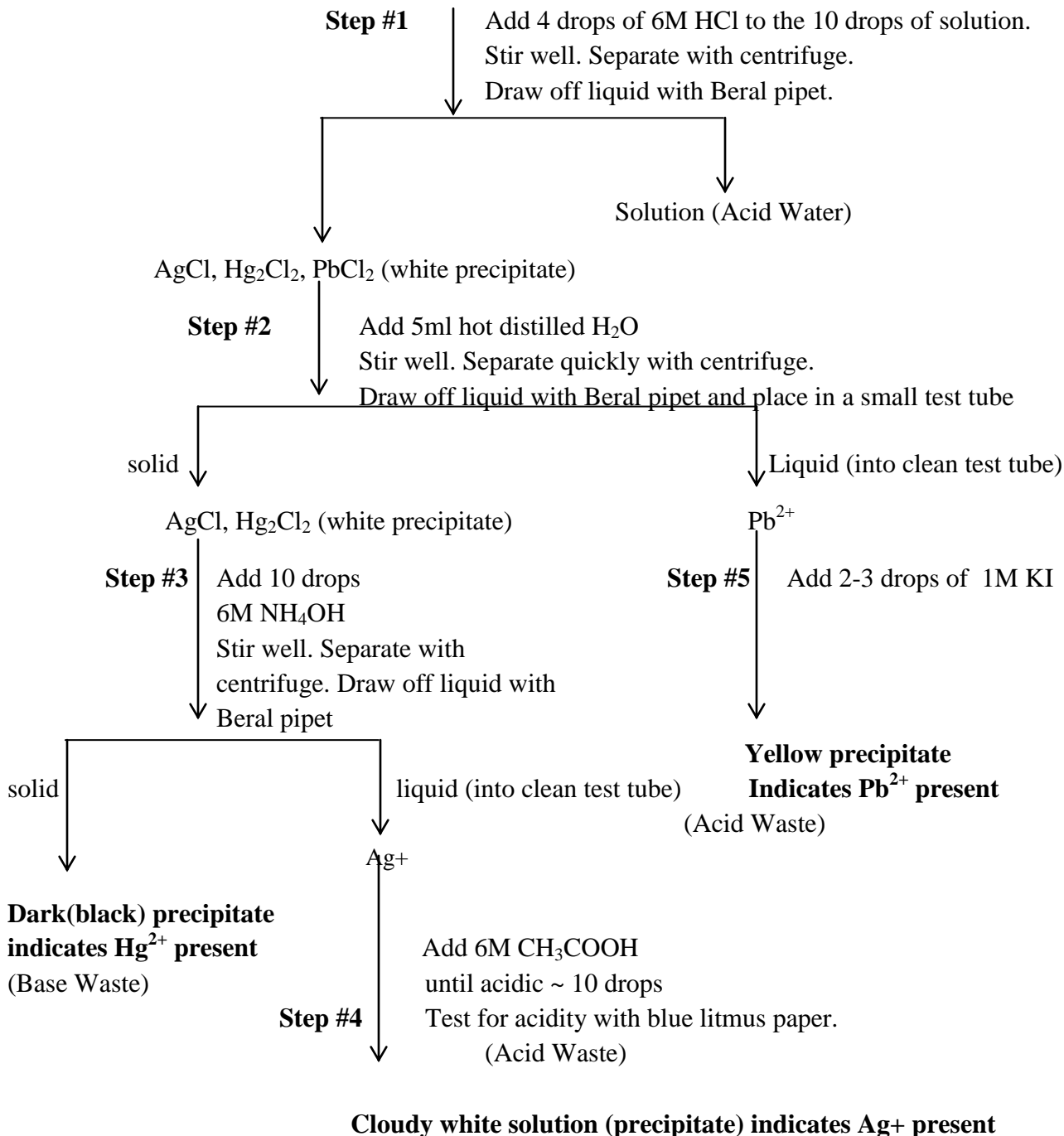
For each cation, one of the tests confirms the presence or the absence of the ion in solution. The test that produced a very dramatic change for one of the ions with little or no change for the others is the confirmatory test for that ion. Briefly summarize the confirmatory test for each ion in the space provided.

Report should state the identity of all cations present in the unknown. Use a **flowchart** to provide the evidence for the presence of the identified cations. Record the cations and record your observations following each experiment in space provided in steps 1-5 on report form.

Flow Chart

(For known and unknown solutions)

Begin with 10 drops of the **Known** solution, which contains **Ag⁺, Hg₂²⁺ and Pb²⁺**, in a small test tube.



Repeat steps 1-5 using 10 drops of assigned Unknown solution in place of Known solution. Pour all waste into appropriate waste containers.

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REPORT SHEET

Name _____

Instructor _____

Unknown # _____

Date _____

PART I. OBSERVATIONS FOR KNOWN SOLUTION:

Step 1: _____

Step 2: _____

Step 3: _____

Step 4: _____

Step 5: _____

PART I. OBSERVATIONS FOR UNKNOWN SOLUTION:

Step 1: _____

Step 2: _____

Step 3: _____

Step 4: _____

Step 5: _____

Unknown Identification

Unknown Number: _____

Ions Present in Unknown: _____

Confirmatory test for your identified ion(s):

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Name: _____

Pre-Laboratory Questions and Exercises

Due before lab begins. Answer in the space provided.

1. Define the following terms.

Qualitative analysis-

Quantitative analysis-

2. Write an ionic and net ionic equation for the following reaction between Pb^{2+} and HCl.

3. A water sample is suspected to be contaminated by Hg(II) ions. How could you determine if the sample is really contaminated?

4. What two things are extremely important to remember when centrifuging?

5. How would you separate Pb^{2+} from Ag^+ in a mixture?

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Name: _____

Post-Laboratory Questions and Exercises

Due after completing the lab. Answer in the space provided.

1. Define the following terms.

Decantation –

Contamination-

Centrifuge-

Supernatant-

2. What reagent(s) may be used to identify and confirm Ag^+ , Hg^{2+} , and Pb^{2+} ions?

3. What are the colors of the following solid precipitates?

AgCl

PbCrO_4

PbI_2

Hg_2O

AgI