# Qualitative Analysis

Chemical analysis can be either qualitative or quantitative in nature. In qualitative analysis we want to know which elements or characteristic chemical species are present. In quantitative analysis we are interested in the relative amounts of the components present. We will be concerned with qualitative analysis in this laboratory. The classical qualitative analysis scheme has been around for well over 100 years, but it continues to be an important part of any chemist's training. It offers an effective means for presenting descriptive inorganic chemistry in the laboratory and it illustrates not only descriptive chemistry, but also important chemical principles, especially those involving ionic equilibria. I hope in the next few weeks that you will gain new insight into these chemical principles.

## Overview

We will be developing an analytical scheme for the following ions:

$$Ag^{+}, Hg_2^{2+}, Pb^{2+}, Cu^{2+}, Hg^{2+}, Cd^{2+}$$

Cations are separated into groups, using group reagents. The ions within a group are then separated from each other. A confirmatory test is carried out for each ion when separations have ensured that interfering ions have been removed. Usually this means isolation from all other cations.

Separations are achieved by selective precipitation. Subsequent separations may involve differential solubility in water, acid, or base, or performing a redox reaction to convert an ion to a different oxidation state. Confirmatory tests often involve a color change or formation of a precipitate characteristic of a particular ion. Confirmatory tests are most often performed on an ion separated from all others, and **not** on the original sample.

## Laboratory Techniques for Semi-micro Qualitative Analysis

1. Distilled water (DW) is used at all times, never tap water.

2. It is essential that all glassware be clean, but not necessarily dry, before use.

3. Care should be taken to label test tubes and beakers in order to avoid confusion.

4. Do not place the reagent dropper tips into the mouth of the test tube. Hold them a little above the mouth to avoid contamination.

5. Estimate volumes (20 drops = 1 ml) whenever possible to save time.

6. **Always** mix solutions thoroughly by flicking the test tube. **Never** shake with your finger over the end.

7. The **approximate** pH of a solution is determined by removing a drop of the solution with a stirring rod and touching it to a piece of universal indicator paper.

8. A 100 or 150 ml beaker of boiling **distilled** water is used to heat solutions in test tubes.

9. To perform a precipitation, add the indicated reagent to your test tube and mix thoroughly. Heat the solution if so directed (this sometimes help to form larger particles). When the precipitation appears to be complete, centrifuge the sample. Before removing the supernatant liquid, add a drop of the precipitating reagent. If the precipitation is incomplete, more precipitate will form in the liquid. If this is the case, add a few more drops, centrifuge, and test again.

10. To centrifuge a sample, place the test tube in one opening, and counter balance with another test tube with a similar volume of solution. Run for about 30 seconds, but this may vary with the nature of the precipitate. Cooperate with the centrifuge as we have only three. The supernatant liquid is usually removed (to another test tube) by decantation. A capillary pipet can also be used. The precipitate must be washed, usually with DW. Add a few milliliters, shake, centrifuge, and discard supernatant liquid. Repeat.

11. **Keep careful records of colors, textures, odors, reactions, etc in your notebook**.

## GROUP 1 CATIONS

$Pb^{2+}$, $Hg_2^{2+}$, $Ag^{+}$

1. Add 10 drops of 0.1 M solutions of the three ions in three separate test tubes. Add 4 drops of 6 M HCl to each. As always, observe the results. Keep in mind that you will write balanced equations, usually the **net ionic**, for any observed reactions throughout these procedures. The equations do not **have** to be recorded in your notebook as you go, but it is a good idea to do so to keep track of the reactions.

2. Add 14 drops of DW to any **washed** precipitates (in step #1) and heat in your water bath.

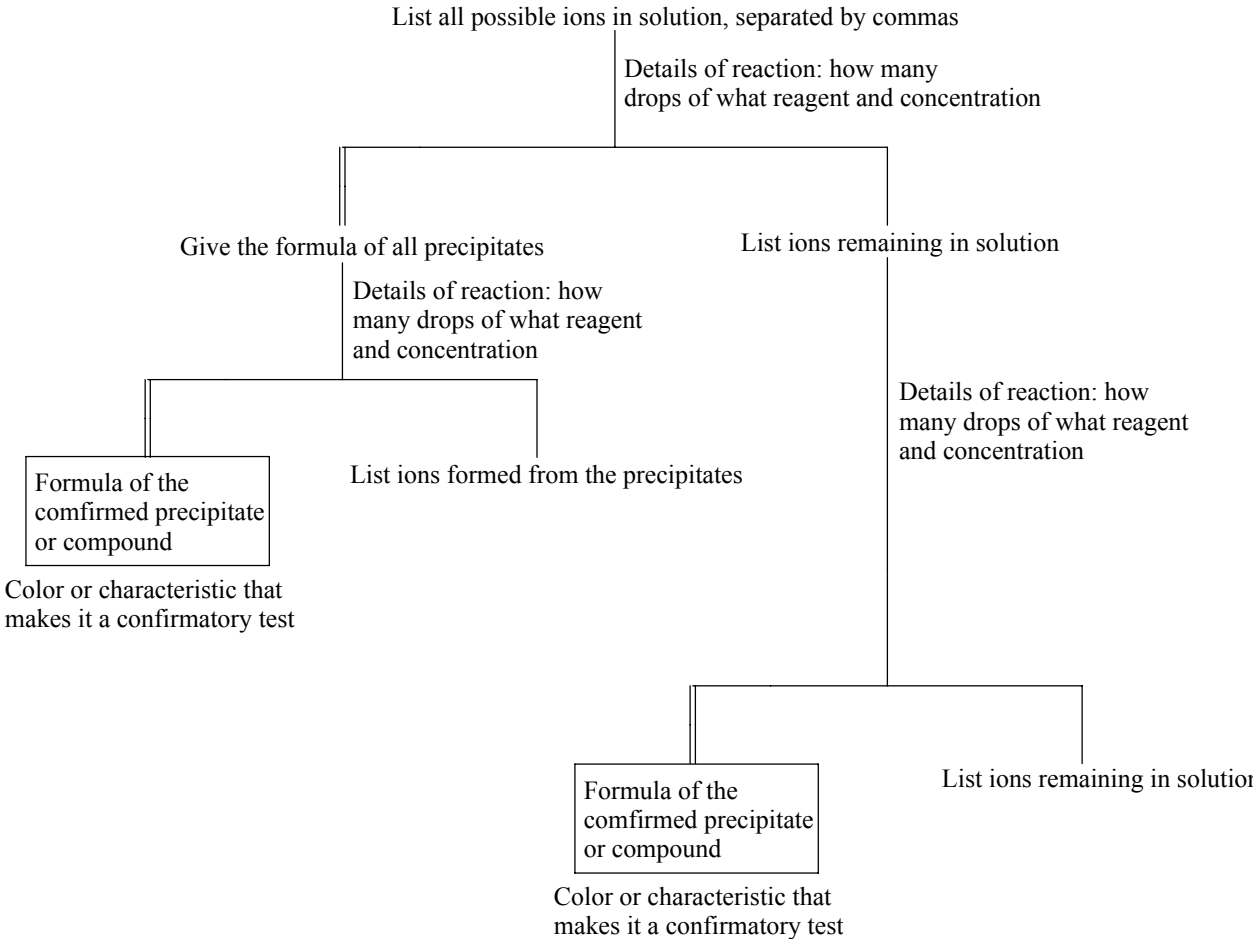
3. To any solutions formed (#2), add 2 drops 6 M acetic acid and 3 drops 1 M $K_2CrO_4$.

4. To any precipitates (#1) which do not dissolve in hot water, add 10 drops of 6 M $NH_3$.

5. Acidify (to test paper) any solutions formed (#4) with 20 drops 6 M $HNO_3$. Make certain the solution is acidic.

The reactions in #3, #4, and #5 are confirmatory tests for that specific ion when it is the only ion possible in solution.

Develop a procedure for separating, and confirming the presence of the three ions in a prepared mixture. This is usually written in the form of a flow chart. Possible ions are shown at the top. Reagents are shown on vertical lines. If a precipitate forms, the precipitate is separated, and indicated on the left with a double vertical line. The supernatant liquid goes on the right with a single line. Confirmatory tests are blocked. **Prepare a mixture of the three ions (about 4 drops of each) and test your procedure.**


List all possible ions in solution, separated by commas
Details of reaction: how many drops of what reagent and concentration
Give the formula of all precipitates
List ions remaining in solution
Details of reaction: how many drops of what reagent and concentration
Details of reaction: how many drops of what reagent and concentration
Formula of the comfirmed precipitate or compound
List ions formed from the precipitates
Color or characteristic that makes it a confirmatory test
Formula of the comfirmed precipitate or compound
List ions remaining in solutio
Color or characteristic that makes it a confirmatory test