General Directions for Qualitative Analysis

In this course students are investigating the properties of selected ions with a view towards discovering ways in which they resemble each other in chemical behavior and also ways in which each ions acts differently from the rest in chemical reactions. This knowledge will allow you to isolate and identify each of these ions.

Some of the tests you perform in qualitative analysis are extremely sensitive. It is important that students use their best techniques at all times to avoid contamination and poor results. Even minute amounts of impurity accidentally introduced into a solution are frequently enough to give a positive test for an ion which should not be present. Correct techniques will help you achieve correct results.

CLEANLINESS:
Cleanliness is of prime importance in avoiding incorrect results in an analysis. Each piece of glassware should be thoroughly washed after each use, and rinsed three times with small amounts of tap water followed by three rinses with distilled water. Keep your wash bottle filled with distilled water for rinsing glassware as well as for diluting solutions. Tap water contains many ions, which can contaminate your work.

CENTRIFUGATION:
In the course of qualitative analysis you will make many separation of precipitates from the solutions which contain them. The standard method of doing this is by filtration but in this course the more rapid procedure of centrifugation is used.

The centrifuge consists of a heavy rotating motor-driven collar mounted on a heavy base containing the mechanical parts of the centrifuge. To use the centrifuge, the solution is placed in a small test tube which is placed into one of the centrifuge tubes. A second counterbalancing tube is placed directly opposite the first. This tube should be filled with water to the same height as the first tube. This counterbalancing procedure is important because the centrifuge revolves so rapidly that if it is unbalanced the resulting vibration could cause serious damage to the machine. After the tubes have been inserted the centrifuge is activated by a switch. Usually 30 seconds are sufficient to settle the precipitate. Do not leave any lab equipment unattended.

PRACTICE EXERCISES:
Record all procedures, observations, and reactions in your laboratory notebook.
1. Place 5 drops of Pb(NO\textsubscript{3})\textsubscript{2} solution in a small test tube, dilute with 20 drops of water, add 2 drops of H\textsubscript{2}SO\textsubscript{4} and mix thoroughly. Centrifuge briefly. Note that the precipitate in this case settles readily. Using a Pasteur Pipet, pipette off all the supermatant liquid (decantate). When pipetting, hold the test tube on a slight angle, depress the bulb of the pipet and place its tip slightly beneath the surface of the liquid. Release the pressure on the bulb slowly to avoid suddenly sucking up the filtrate which might carry with it fine particles of the precipitate. Be careful never to allow the filtrate to enter the bulb of the pipet as this will gradually contaminate the bulb and your reaction. Place the filtrate into a clean test tube,
repeating as often as needed to remove as much of the filtrate as possible. Centrifuge the filtrate again to see if your separation was really complete.

a. To the precipitate in the first test tube, add 10 drops of ammonium acetate solution, warm 6-8 minutes in a hot water bath (use your 250 mL beaker for the water bath and always use distilled water to avoid build-up of mineral deposits in the beaker). At the end of this time, most of the precipitate should be dissolved. Remove the test tube from the hot water bath and immediately add one drop of potassium chromate ($\text{K}_2\text{CrO}_4$). Centrifuge. Would this precipitate or the previous one appear to be a better test for identifying lead ions? Explain your reason.

2. **Completeness of Precipitation.** Whenever an ion is to be removed from a solution by precipitation, one must check to be sure that the ion has been COMPLETELY removed from the solution. The procedure for this is to remove the filtrate from the precipitate, place it in a clean test tube and add to it one to two drops of the reagent being used as the precipitating agent. If no new precipitate forms the precipitation is complete. If more precipitate forms, add 2-3 more drops of the precipitating reagent to the filtrate, re-centrifuge and combine the precipitates with a small amount of distilled water.

a. Place 5 drops of $\text{Cu(NO}_3\text{)}_2$ copper nitrate in a small test tube. Dilute with 1 mL of distilled water. In order to remove the $\text{Cu}^{2+}$ ions from the solution add 3 drops of sodium hydroxide (NaOH). Centrifuge and separate the filtrate from the precipitate. Have you completely removed the copper ions from the solution? How can you tell?

b. Check the filtrate for completeness of precipitation by adding 2-3 more drops of sodium hydroxide. If more precipitate forms, centrifuge and repeat the procedure until the filtrate is free of copper ions.

3. **Washing Precipitates.** During analysis, precipitates must be washed free of the solution from which they formed or many difficulties arise. After centrifuging and removing the filtrate, washing is carried out by stirring up the precipitate with small portions of distilled water, or some other designated reagent and then recentrifuging. Often one or two washings are adequate but as the following example will show, this may not completely remove contaminating ions.

a. Dilute 3 drops of $\text{BaCl}_2$ with a few drops of water, add 4 drops of $\text{K}_2\text{CrO}_4$ and centrifuge. Note that the solution is yellow, showing that an excess amount of $\text{K}_2\text{CrO}_4$ was used. Remove the filtrate and wash the precipitate, which is the $\text{BaCrO}_4$ with 10 drops of distilled water. Remove the wash water to a clean test tube and test it with a drop of silver nitrate. If a precipitate forms in the wash water it indicates the presence of excess chromate ions or excess chloride ions are being removed from the precipitate ($\text{AgCl}$ is white and $\text{Ag}_2\text{CrO}_4$ is red). If a precipitate forms in the wash water, wash the original $\text{BaCrO}_4$ precipitate with a new 10 drop portion of water, centrifuge and remove the second wash and test it with silver nitrate. Continue the washing until no more silver chloride or silver chromate appears in the wash water. How many times did you have to wash the precipitate until you removed all trace of excess chloride or
chromate ions? After which washing did most of the contamination appear to be removed?