DETERMINATION OF AN UNKNOWN CHLORIDE

The determination of a soluble chloride salt concentration is a classic titrametric analysis. A titration involves delivering a measured amount of a solution whose concentration is known accurately (the **titrant**) into a solution whose concentration is not known (the **titrate**). The purpose of the titration is to determine the number of moles of titrate present. When the reaction is complete, some physical change is observed, indicating the **endpoint** of the titration. The endpoint of a titration occurs when stoichiometric ratios of reactants are present and must be determined accurately.

In the titration in this lab, a dilute solution of silver(I) nitrate with a known concentration acts as the titrant. It is added to a salt solution with an unknown amount of chloride, i.e. the titrate. Silver(I) chloride, a white insoluble solid will precipitate from the solution. In order to detect when all the AgCl has been precipitated, another reagent is used as an **indicator**. The indicator in this lab, potassium chromate, is yellow and reacts with silver(I) ions to form a bright orange silver(I) chromate precipitate. This solid is slightly more soluble than the silver(I) chloride so it does not form until essentially all the chloride has precipitated from the solution.

$$Ag^+_{(aq)} + Cl^-_{(aq)} \rightleftharpoons AgCl_{(s)} \text{ at low silver(I) ion concentration}$$

$$2\,Ag^+_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightleftharpoons Ag_2CrO_{4(s)} \text{ at higher silver(I) ion concentration}$$

All standard solutions must first be standardized using a **primary standard** because of potential evaporation. A primary standard is a solid that is stable and does not pick up water. The primary standard in this experiment is purified sodium chloride.

In this lab you will perform six titrations. In the first three titrations you will use a known amount of a pure NaCl sample to determine the exact concentration of an approximate 0.05 M silver(I) nitrate solution. The endpoint is the first permanent orange-red color of Ag_2CrO_4. From this information one can determine the concentration of the $AgNO_3$. The last three titrations will allow you to find the percentage of chloride in your salt when used in conjunction with the average silver(I) nitrate concentration.

Note: Silver is a heavy metal toxin and should never be flushed down the drain. Dispose of all silver waste (silver(I) nitrate and silver(I) chloride) in the waste bottles provided. Silver(I) nitrate is very expensive; each pair of students should use no more than 300 mL of the solution. Do NOT dispose of your silver(I) nitrate solution until you are positive that you are finished with all titrations for parts A and B!

PROCEDURE:

Part A: ***Standardizing the Silver(I) Nitrate Solution***

1. Place about 300 mL of 0.05 M $AgNO_3$, in a clean Erlenmeyer flask and label.

2. Clean a 50 mL buret with soap and water. Use 5 mL of your silver(I) nitrate solution for the final rinse. This silver(I) nitrate solution is the only solution that will be placed in your buret! Fill the buret to the 0.00 mL mark with the $AgNO_3$ solution.

3. Use an analytical balance to weigh three 0.1000 - 0.1200 gram samples of purified NaCl. Record exact mass.

4. Add about 30 mL distilled water to each sample to dissolve. Add 3 drops of indicator (K_2CrO_4).

5. Titrate with 0.05 M $AgNO_3$ solution as you continually swirl the flask to a lovely peach end point. As you add the silver(I) nitrate solution initially in short bursts you will see the orange-red color form and disappear as the solution is swirled. As you approach the end point (which should be between 20-40 mL) the color should begin to persist. At this point you should be adding the solution dropwise. Read the buret to the nearest 0.02 mL. Stop when the sample has a permanent faint peach color.

6. Repeat the titration with the second and third samples.

Part B: ***Determination of Percent Chloride***

1. Obtain an unknown chloride salt and record the ID number in your lab notebook. Use an analytical balance to weigh three 0.1000 - 0.1200 gram samples.

2. To each sample add 50 mL of distilled water and 3 drops of K_2CrO_4 indicator solution.

3. Titrate each sample with the standardized silver(I) nitrate solution as in part A.

4. Do not dispose your $AgNO_3$ solution until you are sure you do not need to repeat any steps!

CALCULATIONS:

For Part A, calculate the molarity of the silver(I) nitrate solution for each titration. Calculate the Parts Per Thousand (PPT) using the "Parts Per Thousand" handout immediately following this lab procedure. If your PPT is greater than 30 for the three trials, consider omitting a deviant molarity value to improve your PPT.

For Part B, calculate the percent chloride. (*Note:* Use the average molarity of $AgNO_3$ as determined in part A.) Average your three percent chloride values and find the PPT. As in Part A, if one trial is quite different from the other two, report data from all three trials, but only average two trials.

POSTLAB QUESTIONS:

1. What is the largest source of error in this experiment? Describe a scenario whereby a percent yield of chloride could be of use in industry. Explain.

2. How would the following hypothetical errors affect the calculated % chloride (increase, decrease or no change)? Explain.
   a. The pure sodium chloride was left open in the scale room and absorbed moisture.
   b. The calculated molarity of the silver(I) nitrate solution was 5% too high.
   c. Two mL of $AgNO_3$ are added beyond the chromate end in titrating the unknown chloride.

3. 35.46 mL of a silver(I) nitrate solution was used to reach the chromate end point with a 50 mL solution containing 0.1165 g of pure NaCl. What is the molarity of the $AgNO_3$ solution?

4. How many mL of the silver(I) nitrate solution used in question 3 will react with 0.2595 g of $BaCl_2$ dissolved in 50 mL of water?

5. A solid chloride sample weighing 0.09969 g required 18.25 mL of 0.05205 M $AgNO_3$ to reach the chromate end point. What is the % chloride in this sample?