CH 221 Fall 2022:
"Unknown Chloride"
(online) ***Lab*** *- Instructions*

Note: **This is the lab for sections 01** *(Veterans Day!)* **and W1 of CH 221 only.**

- *If you are taking section H1 of CH 221*, please use this link:
  http://mhchem.org/s/7a.htm

Step One:

Watch the lab video for the "Unknown Chloride" lab, found here:
http://mhchem.org/w/7.htm
Record the data found at the ***end*** of the lab video on page Ib-7-5.

Step Two:

Complete pages Ib-7-5 through Ib-7-9 using the "Unknown Chloride" video and the actual lab instructions on pages Ib-7-2 through Ib-7-3. Include your name on page Ib-7-5!

Step Three:

Section 01: Submit your lab during lecture at **9 AM on Wednesday, November 16**. A printed copy is required for full credit; emailed labs will invoke a point penalty.

Section W1: **Submit your lab (**pages Ib-7-5 through Ib-7-9 *only* to avoid a point penalty**) as a *single* PDF file to the instructor via email (mike.russell@mhcc.edu) on Wednesday, November 16 by 11:59 PM.** I recommend a free program (ex: CamScanner, https://camscanner.com) or a website (ex: CombinePDF, https://combinepdf.com) to convert your work to a PDF file.

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

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 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 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 ions to form a bright orange silver chromate precipitate. This solid is slightly more soluble than the silver chloride so it does not form until essentially all the chloride has precipitated from the solution.

$$\mathbf{Ag^{+}(aq) + Cl^{-}(aq) \rightleftharpoons AgCl(s)}$$ at **low** silver ion concentration

$$\mathbf{2\,Ag^{+}(aq) + CrO_4^{2-}(aq) \rightleftharpoons Ag_2CrO_4(s)}$$ at **higher** silver 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 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 nitrate concentration.

Note: Silver is a heavy metal toxin and should never be flushed down the drain. Dispose of all silver waste (silver nitrate and silver chloride) in the waste bottles provided.

PROCEDURE:

Part A: ***Standardizing the Silver Nitrate Solution***

1. Clean a 50 mL buret with soap and water, then rinse well with water.

2. Fill your buret with silver nitrate from an amber bottle. To prevent contamination, ***never*** add anything to the amber bottle. Fill the buret to the 0.00 mL mark with the $AgNO_3$ solution. Drain 5 mL from the buret into a beaker (to remove air bubbles) and fill to 0.00 mL again. *Note:* $AgNO_3$ is the only solution that will be placed in your buret! Also, do not dispose of $AgNO_3$ in the sink – place this heavy metal in a waste container.

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

4. Add about 50 mL of distilled water to each sample in a 125 mL Erlenmeyer flask (or larger) to dissolve the NaCl sample. Add about three 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 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 0.01 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 notes. 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 in an Erlenmeyer flask. Titrate each sample with the standardized silver nitrate solution as in part A.

3. When done, place excess $AgNO_3$ in the waste container and rinse the buret with water before leaving the lab.

CALCULATIONS:

For Part A, calculate the molarity of the silver nitrate solution ([$AgNO_3$]) for each titration. Calculate the Parts Per Thousand (PPT) for [$AgNO_3$] using the "Parts Per Thousand" handout in the “Lab Notes” of the Companion. 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 for the %Cl values. 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.

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Determination of an Unknown Chloride

YOUR NAME: ____

DATA: *Watch the video (***http://mhchem.org/w/7.htm***) to acquire these values using the data at the very end:*

<u>Part A</u>

NaCl sample #1 (g): ______________________ mL of $AgNO_3$ #1 (mL): ______________________

NaCl sample #2 (g): ______________________ mL of $AgNO_3$ #2 (mL): ______________________

NaCl sample #3 (g): ______________________ mL of $AgNO_3$ #3 (mL): ______________________

<u>Part B</u>

Unknown sample #1 (g): ______________________ mL of $AgNO_3$ #1 (mL): ______________________

Unknown sample #2 (g): ______________________ mL of $AgNO_3$ #2 (mL): ______________________

Unknown sample #3 (g): ______________________ mL of $AgNO_3$ #3 (mL): ______________________

<u>Part A Calculations</u>: *Determination of an Unknown Chloride*

Show all work, use significant figures and circle the final answer for full credit.

Use the space below to calculate three values for the molarity of the $AgNO_3$ solution. The lab video offers help with this calculation. *Show all work! Watch significant figures!*

Using the three calculated values of [$AgNO_3$] from above, calculate the **average concentration** of $AgNO_3$, the **average deviation** of your molarity calculations and the precision in **parts per thousand**. (The parts per thousand handout can be found here: **http://mhchem.org/ppt**)

<u>Part B Calculations</u>: ***Determination of an Unknown Chloride***

Show all work, use significant figures and circle the final answer for full credit.

Use the space below to calculate three values for the %Cl of your unknown sample. The lab video offers help with this calculation. *Show all work! Watch significant figures!*

Using the three calculated values of %Cl from above, calculate the **average %Cl**, the **average deviation** of your %Cl calculations and the precision in **parts per thousand**. (ppt handout: http://mhchem.org/ppt)

Part C Postlab Questions: *Determination of an Unknown Chloride*

Show all work, use significant figures and circle the final answer for full credit.

1. 35.46 mL of a silver 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?

2. How many mL of the silver nitrate solution used in question 1 above will react with 0.2595 g of $BaCl_2$ dissolved in 50 mL of water?

3. 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?

<u>Part C Postlab Questions</u>: ***Determination of an Unknown Chloride - continued***
Show all work, use significant figures and circle the final answer for full credit.

4. 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.

   This will **increase** **decrease** **not change** the percent chloride. ***(circle one)***

   Explain your answer:

   b. The calculated molarity of the silver nitrate solution was 5% too high.

   This will **increase** **decrease** **not change** the percent chloride. ***(circle one)***

   Explain your answer:

   c. Two mL of $AgNO_3$ are added beyond the chromate end in titrating the unknown chloride.

   This will **increase** **decrease** **not change** the percent chloride. ***(circle one)***

   Explain your answer:

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