AP CHEMISTRY

Silver Chloride Gravimetric Analysis

(115 Points)

Name, Date and Lab Partner (5 Points)

Procedure (20 Points)

Complete, step-by-step account

of actions performed

Data (25 Points)

Observation Table (10 Points)

Reagent Masses (10 Points)

Reagents Volumes (5 Points)

Filter Paper/Crucible Masses (10 Points)

Calculations (25 Points)

Volume of Silver Nitrate (5 Points)

Experimental Yield (1 Points)

% Chloride Ion (5 Points)

Theoretical Yield (5 Points)

Percent Yield (2 Points)

Percent Error (2 Points)

Empirical Formula (5 Points)

Conclusion (25 Points)

Overall Neatness and Organization (5 Points)

Safety (10 Points)

This part is determined by proper lab safety which includes having your goggles and lab coat on properly at all times. Each reminder by the instructor will result in a deduction of 5 or 10 points. This component of the grade can be negative.

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**INTRODUCTION TO ANALYTICAL CHEMISTRY – LAB**

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**EXPERIMENT II**

**GRAVIMETRIC DETERMINATION OF CHLORIDE**

**INTRODUCTION**

For any given analyte, numerous analytical methods are available in the literature, each having unique advantages and disadvantages. The two most common methods for the determination of chloride ion are based on its quantitative reaction with silver ion to form silver chloride. One method involves the isolation of the silver chloride precipitate by filtration, determining its mass, and using stoichiometry to calculate the percent chloride in the sample, this is termed **Gravimetric Analysis**.

Alternately, the chloride ion may be titrated with a standard solution of silver ion and the volume of titrant used to calculate the percent chloride in the sample. This **Volumetric Analysis** is often described as a precipitate-forming titration.

In this experiment you will obtain an unknown sample, containing chloride ion, and you will carry out a gravimetric analysis. Gravimetric methods are among the most accurate and precise methods of quantitative analysis. These advantages are counterbalanced by the fact that they are often time consuming, require considerable attention to detail and are limited to sample size and concentrations which yield a weighable quantity on a conventional analytical balance. While many gravimetric methods have been at least partially replaced by newer, faster techniques, there remain several situations where the only suitable analytical techniques are gravimetric. For these reasons you should become experienced and competent with gravimetric techniques, such as the gravimetric determination of chloride.

**THEORY**

Addition of a solution containing silver ion [usually an aqueous silver nitrate (AgNO3) solution] to a dissolved sample containing chloride ion will quantitatively precipitate the chloride ion as solid silver chloride (AgCl), due to the small solubility product constant of silver chloride (ksp = 1.82 x 10-10). The precipitate may be isolated from solution by filtration, the material dried, weighed and the percent chloride (% Cl-) calculated from the stoichiometry of the reaction:

Ag+(aq) + Cl- (aq) →AgCl(s)

Also, silver chloride is light-sensitive and excessive photodecomposition will produce erroneous results, according to the reaction:



The precipitate becomes violet-purple, due to the presence of finely divided silver metal, and results will be low. If silver ion (Ag+) is present, in addition to the above, the following reaction will also occur as a result of the photodecomposition reaction:



If this reaction predominates, the results will be high. To minimize such errors, it is recommended that unnecessary exposure to light be avoided.

Crystallization technique is also critical during the formation of a precipitate. Poor technique can cause incomplete precipitation or the formation of crystals too small to be sufficiently captured by a filtration device. Typically, optimal crystallization occurs when analyte solutions are combined slowly with continuous stirring. Additionally, it is good practice to heat the combined solution to near boiling, with a stir rod in the solution to prevent rapid/explosive boiling. This helps assure proper crystal formation and complete precipitation.

**PRE-LAB**

1. Write a complete chemical equation that represents a reaction between silver nitrate and your unknown chloride solution (assume the cation is “X2+”).
2. Why does the low ksp of AgCl make it a good species for gravimetric analysis?
3. List 3 possible sources of error to avoid that are specific to this experiment.
4. Document how you will add the silver nitrate solution to your unknown solution, including any relevant equipment usage.

**PROCEDURE**

***Preparation of the Sample for Analysis***

Obtain the unknown sample from your instructor. Measure out about 2g of sample. Prepare an unknown sample solution in a 400mL beaker with a total volume of approximately 150mL. Add approximately 0.5 mL of reagent-grade concentrated nitric acid (HNO3) to the solution.

***Preparation of Precipitating Reagent***

An aqueous solution of 0.5 M silver nitrate is available in the laboratory. The highest ionic charge for any of the unknowns is 3+. Using this information, calculate the volume of the silver nitrate solution required to precipitate all of the chloride ions in the sample (assuming the sample is pure). Include a 10% excess in the final volume to compensate for the fact that the calculation is based on an assumption that could be incorrect.

***Precipitation***

Add the calculated amount of silver nitrate solution to your unknown solution using the technique determined in the pre-lab. Once the solutions are mixed, cover the combined solution with a watch glass and leave the stirring rod in the beaker. Heat the solution, on a hot plate, to nearly boiling to coagulate the silver chloride precipitate. Let each solution cool and settle. Allow the solution to digest (in the dark) overnight.

***Filtration***

Vacuum filter the solution through a crucible/filter paper/funnel assembly. Once transfer is complete, wash the precipitate twice with about 2 mL of 1:500 nitric acid/water solution. Continue to vacuum the precipitate until dry. Carefully remove the crucible from the funnel and place it in the oven to dry in a dark area for 1 hour. Once the filter paper is completely dry, mass the filter paper/crucible assembly.

**DATA ANALYSIS**

1. Re-write the complete chemical equation for the reaction performed per the pre-lab.
2. Calculations
3. Volume (mL) of 0.5M silver nitrate needed to completely react 2 g of XCl? (If X3+).
4. Experimental grams of precipitate collected.
5. Determine the percent composition of chlorine ions (Cl-) in you unknown.
6. Theoretical yield of precipitate in grams. (ask for the identify of your unknown’s cation)
7. Percent yield and percent error for mass of precipitate produced.
8. Try to use your %Cl- data to determine the empirical formula of the unknown. (use the cation identity)

**CONCLUSION**

Write a conclusionary paragraph that highlights:

* The application/theory of gravimetric analysis.
* A discussion of the results of your experiment and how they may or may not have differed from the theoretical outcome.
* Possible sources of error and whether your conclusions confirm the assumption that the chloride reactant was aluminum chloride.
* Ways to improve the experimental procedure in the future.
* Support all of your statements by referencing data, observations, or calculations.