***Experiment 1 Lab Report: Gravimetric Analysis***

**Author:**

**Date:**

**TA Name:**

**This report should be a maximum of 7 pages; you may delete instructions in red and blue font. Font should be 12 point, Times New Roman. Use single-spacing. Do not adjust margins.**

**Save this document with filename “Expt1\_Author\_TAname”, where Author is your name, and TAname is your TA’s name. The filename should not have quotation marks.**

**ABSTRACT and ACCURACY (20 pts)**

***Fill in the blank spaces below in BLUE font.***

In this experiment, gravimetric analysis via precipitation chemistry was used to determine the Al3+ concentration in an *unknown* solution (***Unknown #: 78***).

Average [Al 3+] ± σ is 0.0246 mmol/mL ± 0.0003 mmol/mL.

The chemical reaction that is the basis of this experiment is: Precipitation

The experiment was performed with 8-hydroxyquinoline as the excess reagent, and at constant pH value of 5; these experimental conditions ensured that as much of the 8-hydroxyquinoline stayed in deprotonated form as possible, and that the aluminum would not become tied up in the form of [Al(OH)4]-, which does not precipitate out with this reagent.

**INTRODUCTION(10 pts)**

***Write your introduction below. Do not exceed the allotted space of ~half page (330 words max).***

The purpose of this experiment is to explore the concept of using the mass of a precipitate to determine the concentration of a given ion in a solution of unknown concentration of that given substance. This described method is called gravimetric analysis. Gravimetric analysis is done by taking an excess amount of precipitating agent, or ligand, to force all of once certain species of solute out of solution. A precipitation reaction works by using a soluble ligand to bond with a soluble species of unknown quantity to create an insoluble solid, which can be then weighed. The sample is then filtered and weighed to determine the concentration in the original solution. In this experiment, gravimetric analysis will be used to determine the concentration of Aluminum ions in a solution of Aluminum Nitrate. The precipitation will be carried out with the use of 8-hydroxyquinoline. Gravimetric Analysis is useful because it has applications in determining the concentrations of dissolved metal ions in aqueous solutions, due to their being able to readily form insoluble salts with certain ligands in the correct conditions.

**EXPERIMENTAL (5 pts - Fill in the blanks)**

***The majority of this section has been pre-written for you and can serve as an example of an experimental section for your next lab report (note the grammar, style, etc.). Fill in the blanks in BLUE font. If your procedure deviated from the description below, indicate the change in BLUE font.***

Three filtering ceramic crucibles were cleaned and dried. After cleaning and before drying, the three crucibles were individually labeled. The crucibles (with filter paper) were dried in an oven (at >100°C) for 118 hours, and then allowed to cool for 90 minutes in a desiccator (until they were approximately at room temperature). The crucibles were weighed again and the drying-cooling-weighing process was repeated until the masses were constant to within ± 0.0005 g. Once stable crucible masses were achieved, the ceramic crucible filters were stored in the desiccator.

Three clean 150-mL beakers were labeled. A sample of Al3+ solution of unknown concentration was obtained. An aliquot of the unknown Al3+ solution was quantitatively transferred to each of the three 150-mL beakers using a 25-mL volumetric pipet.

The following description represents a typical procedure for one trial. One of the solutions in a 150-mL beaker was heated to 84.3°C using a Bunsen burner. At this point, approximately 8.0 mL of a solution of the precipitating agent, 8-hydroxyquinoline (Ox), was added. When precipitation began, the solution was stirred with continued heating to redissolve the precipitate. The solution was slowly further heated and a few drops of 6 M HCl were added, if necessary, to fully redissolve the precipitate.

The Bunsen burner was then removed and 2 M ammonium acetate buffer was slowly (i.e., dropwise) added, with stirring, to the hot solution. The buffer was continuously added dropwise until a yellow precipitate formed, which did not redissolve with further stirring. At this point, an additional 20 mL of the 2 M ammonium acetate buffer was added in order to complete the precipitation. The pH of the precipitation solution was checked with litmus paper to confirm that a pH of 5 had been achieved. If the solution remained clear and free of precipitate up to this point, an additional 5-10 mL of the precipitating agent was added and the pH of the solution was checked again.

The precipitate Al(Ox)3 was allowed to form for 46 hours. Once the three samples of Al(Ox)3 precipitate were fully formed, a filtration station (with a trap) was set up and each precipitate sample was separated from its filtrate solution using a labeled filtering crucible (with filter paper). Each of the three 150-mL beakers and its corresponding Al(Ox)3 precipitate was washed with minimal amount of deionized water to fully transfer the precipitate to the filter crucible and wash the accumulated precipitate. The filtrate solutions from all three samples were discarded in an appropriate waste container.

Each of the three Al(Ox)3 samples collected in its filtering crucible was dried at 120-150°C for 7080 minutes, cooled and weighed. The drying-cooling-weighing process was repeated until consistent mass results were obtained for each Al(Ox)3 sample. Once the masses of all three precipitate samples were measured, the concentration of the unknown Al3+ solution for each sample could then be calculated, and the average concentration (along with the σ value) could be determined.

**RESULTS (35 pts total = Data + Observation + Calculations)**

***Write your results below in the appropriate spaces.***

IMPORTANT: The experimental data and observations reported here, in the “Results” section, must also be recorded in your laboratory notebook to be valid. It is unacceptable to rely on memory. Do not attempt to report data/observations that were never recorded in your notebook, and do not add content to your laboratory notebook after the day of the experiment. These acts of falsification and altering of the notebook are violations of academic and scholarly integrity. If you forgot to record information on the day of the experiment, you can indicate: “not recorded in notebook” in the relevant space below.

**RESULTS: Data (10 pts)**

***Fill in the blank spaces below in BLUE font.***

UNKNOWN #: 78

Volume of Al3+ aliquots (*Step #3*): 25.00 mL (± 0.03 mL\*\*)

\*\*Refer to the “Tolerances for NBS Precision Grade Glassware” table provided in Chapter 2 in the CHEM 7L Lab Manual.\*\*

Recorded temperature (°C) of heated solution in *Step #4* during addition of 8-hydroxyquinoline:

|  |  |  |
| --- | --- | --- |
| **Sample #1** | **Sample #2** | **Sample #3** |
| 84.3 | 84.0 | 86.0 |

Recorded color of pH paper in *Step #5* after addition of buffer; state whether the sample was acidic, basic or neutral:

|  |  |  |
| --- | --- | --- |
| **Sample #1** | **Sample #2** | **Sample #3** |
| Orange  Acidic | Orange  Acidic | Orange  Acidic |

**Table 1. Masses of Filtering Crucibles and Precipitates (*Step #6*)**

|  |  |  |  |
| --- | --- | --- | --- |
| **Sample #** | **Mass of Dry Crucible (g)** | **Mass of Dry Crucible + Al(Ox)3 Precipitate (g)** | **Calculated Mass of Dry Al(Ox)3 Precipitate (g)** |
| **1** | 21.9435 | 22.2223 | 0.2788 |
| **2** | 21.9025 | 22.1881 | 0.2856 |
| **3** | 23.0900 | 23.3747 | 0.2847 |

**RESULTS: Observations (5 pts)**

***Describe your observations in the blank spaces below.***

IMPORTANT: Provide detailed observations for each separate trial as documented in your notebook (e.g., color of precipitate and filtrate, volume and/or number of drops of reagents added, unusual incidents, etc.). Do not rely on your memory. If you forgot to record information for one or more trials in your notebook, you can indicate “not recorded in notebook” in the appropriate spaces below.

*Trial #1: (do not report observations for trials #2 or #3)*

* Sample was heated too much, up to 84.3° C.
* The sample was heated accidentally to 93° C on the second heating cycle when dissolving the precipitate
* This one was the first one to enter the crucible
* Small amounts of filtrate stuck to beaker after copious amounts of water were added to clean out the remainder
* The solution in the filter flask was transparent, indicating that little to no filtrate was lost into the flask

*Trial #2: (do not report observations for trials #1 or #3)*

* Sample was overheated to 84.0°C and was not overheated in the dissolution stage before adding the buffer.
* Small amounts of filtrate were left in the beaker even after copious amounts of deionized water was added to scrub out the filtrate
* Solution in the filter flask was transparent, indicating that there was little to no filtrate that got over the filter and into the flask

*Trial #3: (do not report observations for trials #1 or #2)*

* Sample was heated to 86.0° C in initial heating phase
* There was no need for a second heating while dissolving the precipitate
* While filtering, small amounts of the filtrate was left in the filter flask, it was deemed not significant enough to re filter the entire solution.
* Small amounts of filtrate were left in the beaker after filtration after there was an effort made to scrub the filter
* The crucible was knocked over during the final weighing step. None of the filtrate seemed to have come out.

**RESULTS: Calculations (20 pts)**

***The beginning part of this section is started for you. Show your calculations in the blank spaces below. You must utilize an equation editor for this section.***

The determination of the unknown concentration is based upon the balanced chemical reaction below:

1 Al3+(*aq*) + 3 Ox−(*aq*) ⇄ 1 Al(Ox)3 (*s*)

All calculations are based on the following general formula:

A complete calculation for each separate trial is described below.

*Trial #1*:

*Trial #2*:

*Trial #3*:

Calculation of the average [Al3+]: Show your work and report the average [Al3+] in mol/L and in mmol/mL (show the calculation to convert mol/L to mmol/mL). Watch significant figures.

Average [Al3+] = 0.02463 mol/L = 0.02463 mmol/mL

Calculation of the standard deviation, σ: Show your work and report the standard deviation in mol/L and in mmol/mL (show the calculation to convert mol/L to mmol/mL). Watch significant figures.

σ = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ mol/L = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ mmol/mL

**DISCUSSION (20 pts)**

***Write your discussion below in the allotted space of one page (650 words max).***

The discussion of error analysis should be comprehensive (focus on ± σ, not ± 2σ as stated in the manual). Be critical of the experimental procedure and any underlying assumptions regarding the technique and chemistry; are there areas of improvement? A meta-analysis is not required.

This experiment was carried out to determine the concentration of Al3+ ions in a solution of aqueous aluminum nitrate. The experiment utilized concepts from gravimetric analysis to find an unknown concentration of Al3+ based on the amount of precipitated. The sample used in this particular experiment was sample number 78 and the concentration of Al3+ ions was 0.0246 M ± 0.0003 M.

Even though the reported value had 5 decimal places and 4 significant figures, the final value had to be limited to 3 significant figures and 4 decimal places because the error had significant figures starting on the 4th decimal place. The final reported value of concentration of Al3+ is 0.0246 ± 0.0003. This level of precision indicates that there are many measures that could have been taken in order to reduce the error and improve precision. Firstly, the crucibles were put into the oven initially without filters, requiring premature removal for adding filters. Secondly, the fact that only 8.00 ml of 8-hydroxyquinoline was added leaves the possibility that this was not an excess reagent and that there is still more Al3+ that was not accounted for thrown away while the filtrate was being filtered out of the solution. Next, small deposits of the filtrate was left on the beakers after filtration that was not removable with anything other than a hard scrub with soap and running water, which prohibits the filtrate from being added to the crucible. As noted above, one crucible was knocked over during the final weighing, despite no filtrate visibly coming out of the crucible, there is a possibility that some of the precipitate was thrown into the air without notice, possibly affecting the accuracy of the measurement. The pH test strips that were used to measure the pH were very rough and did not tell much about how high or low the pH was because the deep orange color that the test strip displayed represented a pH range of between 4-7, or to put it in simple terms, only stated whether the solution was acidic or basic. All of the above stated errors may contribute to the calculated value being significantly lower than the actual value of concentration.

Certain measures can be taken, if this experiment is repeated, that can and will increase the accuracy and the precision of the experiment. Firstly, the crucibles that are used should have more than one heating and drying cycle, meaning that they spend more cycles in the oven and desiccator, removing even more water and increasing precision. A larger amount of 8-hydroxyquinoline should be added to insure that all of the aluminum ions are precipitated out. Next, better pH test strips or the use of a pH meter would be essential in ensuring stability of the pH in the solution. Cleaner beakers would also assist in preventing the filtrate from sticking to the inside and ensure a higher accuracy.

There was consistency in the quality of the samples as well. They all displayed behavior like precipitate dissolving when hydrochloric acid was added, and maintained a bright transparent yellow (not counting the precipitate). The filtered liquid was not cloudy, indicating a good filtration. This experiment showed how by using a ligand to precipitate out a soluble species, it is possible to determine the concentration of an otherwise unknown concentration of a sample of a known solution.