

Experiment 2 Lab Report: Gravimetric Analysis

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ABSTRACT (10 pts)

In this experiment, gravimetric analysis was used to determine the Al^{3+} concentration in an unknown solution of $\text{Al}(\text{NO}_3)_3$ (*Unknown #: 151*). The concentration of Al^{3+} was determined by forming an insoluble, yellow precipitate of Al^{3+} via complexation with the bidentate ligand 8-hydroxyquinoline (hq). The chemical reaction (i.e. $\text{A} + \text{B} \rightarrow \text{C} + \text{D}$) is: $\text{Al}^{3+} + 3\text{hq} \rightarrow \text{Al}(\text{hq})_3 + 3\text{H}^+$. The experiment was performed with 8-hydroxyquinoline as the excess reagent, and at pH value of 4.5. This pH value ensured the formation of a 90:10 ratio of Hhq^+ to hq. This equilibrium, created as a result of the pH level, serves as the ideal condition for the projection of $\text{Al}(\text{hq})_3$. The average concentration and sample standard deviation based on three trials are: $0.0698 \text{ mol/L} \pm 0.0010 \text{ mol/L}$.

INTRODUCTION (10 pts)

Gravimetric analysis is defined as a method of determining the quantity of an analyte by using its mass. In this experiment, the mass of the dehydrated analyte was used to find the moles of analyte and then the concentration of an Al^{3+} solution was determined through stoichiometric calculations. In this experiment, the goal was to isolate the soluble aluminum in the unknown solution. To do this, a soluble compound was used to bind to aluminum and then precipitate out of solution. 8-hydroxyquinoline is a ligand that binds to the soluble aluminum, turning it into an insoluble compound. This creates a precipitate in the solution, allowing us to extract all the aluminum from the solution. However, the pH of this reaction is very important. 8-hydroxyquinoline has three different forms it takes, depending on the pH of the solution it is in. At low pH, below 5, 8-hydroxyquinoline forms a positively charged protonated solution (Hhq^+). This protonation creates a positive charge for the molecule, making it soluble. However, it is unable to bind to Al^{3+} because the lone pairs on the nitrogen are taken up by a hydrogen, meaning it is not a suitable variation of the molecule. From pH 5 to 10 it is uncharged and insoluble (hq). The molecule deprotonates, losing the hydrogen bonded to the nitrogen, restoring the nitrogen's formal charge to zero, causing the molecule to be neutral. This neutrality leads to the molecule being insoluble at this pH range. This means that around this neutral pH range, the ligand will precipitate without bonding to the aluminum first, leaving precipitate that does not have aluminum. Above pH 10, the molecule is ideal, able to bind to Al^{3+} , and is negatively charged due to deprotonation of the alcohol group (hq^-). This means the molecule is soluble, but at this range, the aluminum begins to precipitate on its own. It forms Al biproducts like Al_2O_3 , $\text{Al}(\text{OH})_3$, $\text{Al}(\text{OH})_4$. However, there is a range between 4 and 6 that consists of protonated 8-hydroxyquinoline and deprotonated 8-hydroxyquinoline in constant equilibrium, allowing the deprotonated 8-hydroxyquinoline to bond to the aluminum, forming the precipitate. The pH is very important in this experiment because it changes the protonation of the solution, which changes the charge of the solution, which ultimately changes the binding properties of the solution to the Al^{3+} .

EXPERIMENTAL (23 pts)

Three filtering ceramic crucibles were cleaned, labeled, and dried. The crucibles (with filter paper) were dried in an oven (at $>100^{\circ}\text{C}$) for 96 hours, cooled in a desiccator, weighed, and returned to the desiccator for storage. Three clean 150-mL beakers were labeled. A sample of Al^{3+} solution of unknown concentration (unknown number 151) was obtained. An aliquot of the unknown Al^{3+} solution was quantitatively transferred to each of the three 150-mL beakers using a 10-mL volumetric pipet, with an additional 15-mL of DI water.

The total number of trials was 3. The following description refers to the first trial. The volume of Al^{3+} aliquot was 10.00 mL. This aliquot was placed in a 150-mL beaker which was heated to 75.0°C using a hot plate. Next, 8.00 mL of a solution of the precipitating agent, 8-hydroxyquinoline (Hhq), was added. Precipitate appeared approximately 1 second after addition of Hhq. The visual appearance of the precipitate and liquid were as follows: the liquid quickly turned yellow and cloudy.

After the appearance of precipitate, the solution was stirred and 14 drops of 6 M HCl were added to fully redissolve the precipitate. The total stirring/heating time to redissolve the precipitate was 3 minutes. After dissolution, the visual appearance of the liquid solution was as follows: transparent dark yellow throughout.

The sample was removed from the hot plate and 2M ammonium acetate buffer was added dropwise while stirring the hot solution. The buffer was added until a precipitate formed and did not redissolve with further stirring. A total of 22 drops (or 1.10 mL) were added until the precipitate did not redissolve. The visual appearance of the precipitate and liquid were as follows: it became a clear yellow solution with a few small clumps of light-yellow precipitate floating in solution.

At this stage, an additional 10.00 mL of the 2 M ammonium acetate buffer was added in order to complete the precipitation. The pH of the precipitation solution was 4.0 as determined by pH paper, which turned a color of green.

The same procedure was followed for the 2nd and 3rd trials (see trial-specific details in Table 1). For all trials, the precipitate $\text{Al}(\text{hq})_3$ was allowed to form for 48 hours. A filtration station (with a trap) was set up. The 150-mL beakers with $\text{Al}(\text{hq})_3$ precipitate were washed with minimal amount of deionized water to fully transfer the precipitate to the filter crucibles and wash the solid. The three $\text{Al}(\text{hq})_3$ samples were dried in a $>100^{\circ}\text{C}$ oven for 48 hours. The visual appearance of the solid (precipitate and liquid (filtrate)) were as follows: The precipitate looked cracked and very dry yellow chalky texture while the liquid in the flask looked clear and dark yellow.

Table 1. Analogous experimental details for the 2nd and 3rd trials

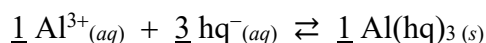
Trial 2	Temperature of solution	75.0°C
	Volume of Hhq added	8.00 mL
	Time for precipitate to appear after Hhq	0 seconds
	Visual appearance of precipitate <u>and</u> liquid after addition of Hhq	Cloudy yellow
	Drops of 6M HCl added	15
	Time to redissolve the precipitate	3 minutes
	Visual appearance of liquid after redissolving the precipitate	Transparent yellow
	Drops / volume of buffer initially added	12 drops, 0.60 mL
	Visual appearance of precipitate <u>and</u> liquid after addition of buffer	liquid was dark yellow and there were a few precipitate pieces floating in solution
	Additional volume of buffer added	10.00 mL
	Final pH of solution, color of pH paper	4.5, green

Trial 3	Temperature of solution	75.0°C
	Volume of Hhq added	8.00 mL
	Time for precipitate to appear after Hhq	seconds
	Visual appearance of precipitate <u>and</u> liquid after addition of Hhq	Cloudy yellow
	Drops of 6M HCl added	14
	Time to redissolve the precipitate	3 minutes
	Visual appearance of liquid after redissolving the precipitate	Clear yellow
	Drops / volume of buffer initially added	20 drops, 1.00 mL
	Visual appearance of precipitate <u>and</u> liquid after addition of buffer	Clear deep yellow with a few small bits of light yellow precipitate floating
	Additional volume of buffer added	10.00 mL
	Final pH of solution, color of pH paper	4.5, green

RESULTS (Data + Calculations) 20 points**Table 2. Masses of Filtering Crucibles and Precipitates (Step #9)**

Trial #	Mass of dry crucible (g)	Mass of dry crucible + Al(hq)₃ (g)	Mass of dry Al(hq)₃ (g)
1	19.7833	20.0993	0.3160
2	20.1316	20.4521	0.3205
3	21.2147	21.5396	0.3249

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



All calculations are based on the following general formula:

$$[\text{Al}^{3+}] = \frac{\text{mass of Al(hq)}_3 \text{ precipitate (g)}}{\text{molar mass of Al(hq)}_3 \text{ precipitate } \left(\frac{\text{g}}{\text{mol}} \right)} \times \frac{1 \text{ mol Al}^{3+}}{1 \text{ mol Al(hq)}_3} \times \frac{1}{\text{volume of Al}^{3+} \text{ solution (L)}} = \text{_____}$$

A calculation of $[\text{Al}^{3+}]$ for each trial is described below.

Trial #1:

$$[\text{Al}^{3+}] = \frac{0.3160 \text{ g}}{459.42 \text{ g/mol}} \times \frac{1 \text{ mol Al}^{3+}}{1 \text{ mol Al(hq)}_3} \times \frac{1}{0.01000 \text{ L}} = 0.06878238 \text{ M} = \mathbf{0.06878 \text{ M}}$$

Trial #2:

$$[\text{Al}^{3+}] = \frac{0.3205 \text{ g}}{459.42 \text{ g/mol}} \times \frac{1 \text{ mol Al}^{3+}}{1 \text{ mol Al(hq)}_3} \times \frac{1}{0.01000 \text{ L}} = 0.06976187 \text{ M} = \mathbf{0.06976 \text{ M}}$$

Trial #3:

$$[\text{Al}^{3+}] = \frac{0.3249 \text{ g}}{459.42 \text{ g/mol}} \times \frac{1 \text{ mol Al}^{3+}}{1 \text{ mol Al(hq)}_3} \times \frac{1}{0.01000 \text{ L}} = 0.0707196 \text{ M} = \mathbf{0.07072 \text{ M}}$$

Calculation of the average $[\text{Al}^{3+}]$:

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$$

$$\bar{x} = \frac{0.06878238 M + 0.06976187 M + 0.0707196 M}{3}$$

$$\bar{x} = 0.06975453 M = \mathbf{0.0698 M}$$

Calculation of the sample standard deviation:

$$s = \sqrt{\frac{\sum_1^n (x_i - \bar{x})^2}{n - 1}}$$

$$s = \sqrt{\frac{(0.06878238 - 0.06975453)^2 + (0.06976187 - 0.06975453)^2 + (0.0707196 - 0.06975453)^2}{3 - 1}}$$

$$s = 0.00096882 = \mathbf{0.0010}$$

Calculation of the % relative standard deviation:

$$\%RSD = \frac{s}{\bar{x}} \times 100\%$$

$$\%RSD = \frac{0.00096882}{0.06975453} \times 100\%$$

$$\%RSD = 1.38890124\% = \mathbf{1.4\%}$$

DISCUSSION (30 pts)

The calculated average concentration of the sample number 151 of Al^{3+} solution is 0.0698 M and the standard deviation among the three trials is 0.0010 M. The calculated %RSD is 1.4%. This %RSD is good and indicates a precise data set. This suggests that there was little random error in the experiment. The most important action that led to a higher degree of precision in this experiment was consistency throughout. All the trials were completed following the same procedure and under the same conditions. The same equipment was used for all trials. The scale used was the same and all the glassware used was constant as well. This consistency is what led to the relatively high degree of precision for this experiment.

One experimental assumption that was in this experiment is assuming the precipitate was completely dry. The sample was dried by first vacuuming the water out of the precipitate to forcefully extract and liquid. The samples were then placed in an oven for 48 hours to evaporate off the rest of the water in the sample. However, it is not certain that the sample was completely dehydrated. It is assumed that the sample only consisted of precipitate for the sake of convenience in this experiment. If there was water still present in the sample, it would have thrown off the mass of the sample, which in turn would have led to an artificially high calculated Al^{3+} concentration. Another experimental assumption that was present was assuming that all the Al^{3+} in solution reacted with the 8-hydroxyquinoline. While the solution reacted for a long time, it is not certain that the reaction was run to completion. If the assumption was untrue this could lead to a lower calculated value of the mass that it should have been. This would have led to a lower calculated Al^{3+} concentration. A third assumption in this experiment that may have led to error if it was untrue was if the 8-hydroxyquinoline reagent was too much in excess. It is known that the reaction in excess in this experiment was the 8-hydroxyquinoline, but the aluminum concentration varies, meaning the ratio of each reagent is variable. If there is too much excess of 8-hydroxyquinoline for our sample, it will precipitate on its own, contaminating the $\text{Al}(\text{hq})_3$ precipitate. This would lead to a higher mass of the precipitate, leading to an increase in calculated concentration of Al^{3+} .