Sample Lab Report: Gravimetric Analysis of Chloride in Solution

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1   Introduction

Gravimetric analysis is an important analytical chemistry technique that is used to identify and quantify the presence of a specific constituent in an unknown sample. The main idea is that a specific constituent is isolated from the unknown and reformed into a known compound via precipitation or volatiliziation methods.[1] If we then know the weight of our final product (precipitate or gas), we can determine the number of moles, and therefore mass of our desired constituent in our initial unknown. Knowing then, the initial weight of our unknown we can the determine the percentage by mass of our constituent in the unknown.

> It is generally a good idea to discuss the main points/more general ideas first, then go to specifics of the experiment later in the intro

In this study we aim to investigate the percentage of chlorine in an unknown, water-soluble salt, denoted M_xCl_y, where M is an unknown, pure cation. Using the % chlorine as well as the fact that M is a pure cation coming from the first 3 groups, we can then predict what our unknown salt is. Dissolving our unknown into a water solution will cause the chlorine and unknown cation to dissociate.

> A very brief description of what we will do, but not describing the lab procedure

$$M_xCl_{y(s)} \longrightarrow xM_{(aq)}^{n+} + yCl_{(aq)}^- \tag{1}$$

Addition of an excess $AgNO_3$ of our solution will cause the formation of $AgCl_{(s)}$, a white precipitate.[2] We choose to form AgCl since it is sparing soluble in water $(0.0014 \text{ g/L at } 20° \text{ C})$[3] and therefore all of the chlorine should fall out of solution. Our overall (unbalanced) reaction is given by

$$M_xCl_{y(aq)} + AgNO_{3(aq)} \longrightarrow AgCl_{(s)} + M_\alpha(NO_3)_{\beta(aq)} \tag{2}$$

Then, if we know the final mass of AgCl, m_{AgCl}, we can determine the number of moles of Cl by

$$n_{Cl} = m_{AgCl} \times \frac{1 \text{ mole AgCl}}{143.32 \text{ g AgCl}} \times \frac{1 \text{ mole Cl}}{1 \text{ mole AgCl}} \tag{3}$$

The number of moles of Cl can be converted to a mass by multiplying by the molecular weight of chlorine

$$m_{Cl} = n_{Cl} \times \frac{35.453 \text{ g Cl}}{1 \text{ mole Cl}} \tag{4}$$

If we knew the initial mass of the unknown we started with, $m_{M_xCl_y}$, and assume a full conversion of all chlorine to AgCl, then the percent chlorine in our unknown, by mass, is given by

$$\%Cl = \frac{m_{Cl}}{m_{M_xCl_y}} \times 100\% \tag{5}$$

Using the %Cl we measure in our unknown, we can then make an educated guess of what our unknown cation, M is and therefore determine the unknown salt M_xCl_y.

Walk the reader through the math/theory of how exactly we can determine the % Cl from this experiment and introduce the more general equations.

Make sure that equations are properly formatted!

2 Experimental Procedure

The procedure which was followed can be found in the laboratory manual.[3]

Note that a balance of 0.01 precision was used instead of the requested 0.0001.[3]

> You should cite the lab manual here

> Only deviations from the lab manual should be reported

3 Data Analysis

Our raw data and percent chlorine for each trial is summarized in Table 1. The average percent chlorine in the unknown is 62%. Sample calculations can be found in section A.2.

> Use tables to summarize raw data and results. Graphs are also great if appropriate.

Table 1: Percent chlorine in each trial. m_i is the initial mass of the crucible, m_f is the final mass of the crucible (with preciptate AgCl).

	$m_{M_xCl_y}$ [g]	m_i [g]	m_f [g]	m_{AgCl} [g]	% Cl
Trial 1	0.25	31.51	32.12	0.61	60.%
Trial 2	0.26	32.21	32.87	0.66	63%

> Should summarize all results in this section. Do not leave results in the sample calculation section (becomes hard to track down!)

In order to determine the unknown salt, we observe first that the overall charge must be neutral. Therefore M should have a positive charge. Second, the mass ratio of M to Cl in the compound should be roughly 2:3, per the 62% Cl by mass we computed. Knowing that the molar mass of Cl is 35, and consider Na has a molar mass of roughly 22, about 2/3 that of Cl, it makes a reasonable first guess. Hence if we assume our salt is then NaCl, then the percent chlorine would be

> Can include the sample calculations here, but I prefer them at the end so they do not clutter this section.

$$\frac{35.45 \text{ g Cl}}{58.44 \text{ g NaCl}} \times 100\% = 60.66\% \tag{6}$$

> Depending on what is requested, you may need to include some reasoning for how you came to some conclusion/result

This is extremely close to our experimentally determined % Cl. If we consider other cations from group 2 and 3 on the periodic table, we find that none of those salts make any sense (work not shown); hence, we can conclude that our experiment suggests that the unknown salt is NaCl.

4   Discussion

In this experiment, we analyzed an unknown water-soluble salt using a precipitation based gravimetric analysis. We determined that the average percent Cl by mass was 62% and that this data suggests that the unknown salt is NaCl (Table 1). Using pure NaCl as a reference, the percent Cl by mass should be 60.66%, yielding a 2% relative error.

While our experimental value is very close to the theoretical value for NaCl, there was still a relatively large error for gravimetric analysis, which should have precision around 0.1 to 0.3 %.[3] Further, we do not know definitely that the starting salt was NaCl, and it could be that large experimental errors have causes us to reach the wrong conclusion. There a variety of sources of errors within the experiment which could have contributed to these differences and/or wrong conclusion. Firstly, the balance we used was only precise to the 0.01 position forcing our measurement of 0.25 g of unknown salt to have a high relative error. Using a more precise balance, such as the suggested analytic balance which has precision to the 0.0001 position,[3] would decrease our uncertainty and increase our final accuracy. Regardless of our initial measurement, a huge source of error can come from not knocking all of the chlorine out of solution and forming our AgCl. All of the precipitate should have fallen to the bottom of the beaker and the solution should have been clear; however, in our case we observed a slightly cloudy solution. Thus it is possible that there was still some chlorine in solution which had not reacted with the silver. This could cause a substantial deviation from the theoretical percent chloride as our final measurements would yield a smaller precipitate mass, and therefore a small percent chlorine in the unknown. Though, it is possible that this error was offset by the error of not fully drying our precipitate. Since our precipitate was formed in water, water was still present in the precipitate, which had a

Always summarize the major results and findings of the experiment at the beginning of the discussion. Try and compare to theoretical/literature values if appropriate.

Should always include an analysis of the sources of errors/approximations in the lab. Nothing is perfect experimentally and there should always be deviations from theoretical values.

cakey structure to it. While we dried our sample in the oven to remove most of the water, leaving it overnight would be best to ensure all of the water was removed. Presence of water in our sample can artificially increase the final mass of our precipitate, leading us to deduce that our unknown sample had a higher percentage of chlorine.

While gravimetric analysis is useful for analyzing substances with high concentration of ions, it fails to be effective at low concentrations. In general, it is a macroscopic technique and a minimum of 10 mg of precipitate is required for effective analysis.[3] First, most analytical balances can only measure to the 0.0001 g which corresponds to 0.1 mg precision so our relative error of uncertainty for weighing will increase as we go to smaller and smaller sample sizes. Additionally, when working with small samples, any loss of product, either through transferring or not getting a full theoretical yield from the reaction, will have a larger relative error than if we had used a larger sample. For example, if we consider theoretical yields of (a) 1000 mg and (b) 5 mg, and a loss of 2mg of precipitate during transferring to the crucible occurred, we have a relative error of 0.2% for (a) and 40% for (b). Hence, using gravimetric analysis for samples with low concentrations can yield high imprecisions and a different method is required to analyze such compounds. Methods such as micro X-ray fluorescent spectroscopy or energy dispersive x-ray spectroscopy should be used instead.

Notice how in analyzing the error, it is discussed how the error will affect the final result

in this case, I answer the first discussion question in text, but will leave the math discussion question for the appendix section. This is a preference.

A   Appendix

A.1   Lab Manual Questions

A.1.1   Question 2

We first determine the percentage of Cl in the unknown. First we determine
the number of Cl moles via Eq. 3

Need to show your work to receive full credit.

$$0.523 \text{ g AgCl} \times \frac{1 \text{ mole AgCl}}{143.32 \text{ g AgCl}} \times \frac{1 \text{ mole Cl}}{1 \text{ mole AgCl}} = 3.65 \times 10^{-3} \text{ moles Cl}$$

Then we can determine the mass via Eq. 4

$$3.65 \times 10^{-3} \text{ moles Cl} \times \frac{35.453 \text{ g Cl}}{1 \text{ moles Cl}} = 0.129 \text{ g Cl}$$

Then the percentage is given by Eq. 5

$$\frac{0.129 \text{ g Cl}}{0.323 \text{ g Unknown}} \times 100\% = \boxed{40.1\%}$$

We now consider the follow scenarios to determine M in the equation $M_x Cl_y$.

a) Let $x = y = 1$. Then $n_{Cl} = n_M = n_{MCl}$. Hence we have that

$$3.65 \times 10^{-3} \text{ moles Cl} \rightarrow 3.65 \times 10^{-3} \text{ moles M}$$

Thus we see that

$$m_M = 0.323 - 0.129 \text{ g} = 0.194 \text{ g}$$

Hence

$$MW = \frac{0.194 \text{ g M}}{3.65 \times 10^{-3} \text{ moles M}} = \boxed{53.2 \text{g mole}^{-1}}$$

b) Let $x = 1$ and $y = 2$. Then $n_{\text{Cl}} = 2n_{\text{M}}$, hence we have that

$$3.65 \times 10^{-3} \text{ moles Cl} \rightarrow 1.83 \times 10^{-3} \text{ moles M}$$

and we still have $m_{\text{M}} = 0.194$ g. Therefore

$$MW = \frac{0.194 \text{ g M}}{1.83 \times 10^{-3} \text{ moles M}} = \boxed{106\text{g mole}^{-1}}$$

c) Let $x = 2$ and $y = 4$. Then $2n_{\text{Cl}} = 4n_{\text{M}} \Rightarrow n_{\text{Cl}} = 2n_{\text{M}}$. So we are in the same situation as above and can quickly see that

$$\boxed{MW = 106\text{g mole}^{-1}}$$

A.2   Sample Calculations

We use trial 1 for all sample calculations. The average value is determined using both trials.

Only 1 sample calcuation for each calculation is needed. Inclusion of more than that will result in reduction of points.

A.2.1   Mass of Precipitate

$$m_f - m_i = 32.12 \text{ g } - 31.51 \text{ g } = 0.61 \text{ g AgCl}$$

A.2.2   Moles of Cl

Using Eq. 3

$$n_{\text{Cl}} = .61 \text{ g AgCl} \times \frac{1 \text{ mole AgCl}}{143.32 \text{ g AgCl}} \times \frac{1 \text{ mole Cl}}{1 \text{ mole AgCl}} = 4.3 \times 10^{-3} \text{ moles Cl}$$

A.2.3 Mass of Cl

Using Eq. 4

$$m_{\text{Cl}} = 4.3 \times 10^{-3} \text{ moles Cl} \times \frac{35.453 \text{ g Cl}}{1 \text{ moles Cl}} = 0.15 \text{ g Cl}$$

A.2.4 Percentage of Cl

Using Eq. 5

$$\frac{0.15 \text{ g Cl}}{0.25 \text{ g M}_{\text{x}}\text{Cl}_{\text{y}}} \times 100\% = 60.\%$$

A.2.5 Average Percent Cl

We find the average of some quantities N quantities x_i to be

$$\langle x \rangle = \frac{\sum_i^N x_i}{N} \tag{7}$$

Thus, a direct computation leads to

$$\langle \%\text{Cl} \rangle = \frac{60 + 63}{2} = 62\%$$

References

(1) Erdey, L. *Gravimetric Analysis: International Series of Monographs on Analytical Chemistry, Vol. 7*; Elsevier, 2013; Vol. 7.

(2) PubChem Compound Summary for CID 24561, Silver chloride. `https://pubchem.ncbi.nlm.nih.gov/compound/Silver-chloride`.

(3) Zhao, M.; Dragisich, V. *General Chemistry Experiments*; MacMillan Learning, 2018.

References follow ACS citation style