Gravimetric Analysis Formal Report

CHEM 1001

Purpose:

The main objective of this experiment is to discover the specific amount of chloride quantity in an unknown soluble salt using gravimetric analysis.

Theory:

Specific substances are very insoluble, to the point where they can be precipitated and their amount can be known, by means of a precipitation reaction. This means we can use gravimetric analysis to determine the percentage of Chloride ions present in a solution using the mass of the solution. Aqueous silver ions and chloride ions bond together to form silver chloride precipitate in the solid state:

$$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl_{(s)}$$

This formula is based on the fact that the reaction goes to completion, since AgCl is highly insoluble. AgCl does exhibit solubility to some extent, which is represented by a small solubility product, Ksp. The Ksp value dictates the degree to which a compound will disassociate in water (high value = soluble, and vice versa):

$$AgCl_{(s)} \rightarrow Ag^{+}_{(aq)} + Cl^{-}_{(aq)}$$

$$K_{sp} = [Ag^{+}_{(aq)}][Cl^{-}_{(aq)}] = 1.6 \times 10^{-10}$$

This low value Ksp is considerably low and is insignificant. This means that once the precipitate is formed only a very tiny portion can dissolve back to the solution.

Once a soluble salt containing silver is added to the Cl^- solution , the silver instantly creates a bond with the chloride ion and forms AgCl until all the Cl^- has been used up. based on the amount of extra silver ion has been added to the solution, the amount of salt left in the solution will vary.

Because the precipitation occurs quickly, the silver chloride will precipitate as a colloid instead of forming proper crystals. The solution is heated and stirred with nitric acid to improve coagulation, so that it cannot pass through the filter paper. Nitric acid is also used to prevent the crystals from turning back into a colloidal state (peptization). If the peptization occurs, the AgCl precipitate will pass through the filter, which is not desirable.

The high speed of the precipitation causes some undesired ions to be trapped in the colloidal residue. For this reason, precipitation should occur at a slow pace, in an acid media, to ensure there is no intervention from anions. If the salt was precipitated in a neutral medium, the anions would co-precipitate with silver. Excess silver ion should be added moderately; when a desirable amount is added, it lessens the solubility of the silver chloride, however too much excess silver ion

causes co-precipitates. Ions that can co-precipitate with chlorine are mercury and lead(II). If one of these ions precipitate with chloride, it will lead to a higher result:

$$Hg^{+}_{(aq)} + Cl^{-}{}_{(aq)} \rightarrow HgCl(s)$$

Another factor that affects the amount of precipitate in the solution is whether the solution was exposed to light or not. AgCl decomposes in the presence of light , which will decrease the amount of Cl^{-} in the precipitate:

$$AgCl_{(s)} \rightarrow Ag_{(s)} + 1/2\ Cl_{2(g)}$$

The precipitate attains a violet color caused by grouping of separated silver. When this photodecomposition happens in the presence of air, the results will turn out to be low , because chlorine gas will be released. When the photodecomposition happens in the presence of excess silver ion in aqueous solution, another reaction occurs:

$$3Cl_{2(g)} + 9H_2O_{(l)} + 5\ Al^{+}_{(aq)} \rightarrow 5AgCl_{s)} + ClO_3^{-}{}_{(aq)} + 6H_3O^{+}_{(aq)}$$

This reaction will cause the amount of AgCl to increase and will lead to higher results, because chlorine gas is recombining with the solid silver to produce AgCl.

The mass of precipitate lost by washing with 100ml of distilled water can be found by multiplying the Ksp in ml of AgCl by 100ml to find the total number of moles in the solution then dividing that number by the molar mass of AgCl:

(1.6 x 10^{-10}mol/L) x (1L/1000ml) x (100ml) = 1.6 x10^{-11}mol

(1.6 x10^{-11}mol) x (143.32 g/mol) = 2.3 x $10^{-9}g$

Procedure:

On each table, an unknown salt was provided. The code number (#361) was recorded. 0.1485g of unknown salt was weighed by difference with an analytical balance in a 250 ml beaker. The volume of 0.1M of $AgNO_3$ was calculated using [(mass of the sample %Cl)/35.5]/0.1], after that the results were converted into ml and 5ml were added to make sure that all the chloride was precipitated. This calculation yielded 23.007ml of $AgNO_3$. 100 ml of distilled water and 1 ml of dilute (6M) HNO_3 was added to the salt. The solution was stirred with a glass stirring rod until the salt completely dissolved. Slowly, the calculated amount of $AgNO_3$ was added to this solution and stirred. The solution was heated on a hot plate, while being stirred until the solution is almost boiling. Completeness of precipitation was checked by adding drops of silver nitrate into the liquid. No additional silver chloride appeared, which means the precipitation was complete. The mixture was moved to a dark place; the drawer. Filter paper was given by the TA and placed in a 50 ml beaker and weighed with an analytical balance. The value was recorded to be 29.4316g. the filtration apparatus was already set up, so the filter paper was placed onto the Buchner Funnel and the funnel placed onto

filtration flask. The filter paper was wet to ensure it adhered to the funnel. The mixture was removed from the drawer and decanted through the funnel. Then 0.01M HNO_3 was added to the precipitate in the beaker. The washings were then decanted through the filter once more. This process was repeated until 60 ml of 0.01M HNO_3 was added, while transferring the precipitate and washings onto the filter. To check if the solution is free of silver ions, small amount of washings was collected in a beaker and tested by adding a few drops of HCL. The washing was complete since no turbidity was observed. The washings were emptied from the filter flask and the precipitate was washed with 5ml of acetone. The filter paper was removed and placed in the 50 ml beaker. The beaker was placed in a 95C oven for 15 minutes, then was removed and cooled for 5 minutes. The final weight of the beaker was weighed with an analytical balance and was obtained to be 29.7074g. this meant the dried sample mass was 0.2758g. lastly the accepted value was gotten from the TA to be 55.16% of Cl

Observations:

Sample number: 361

Accepted value(%Cl): 55.16%

Table 1 – Data of Original and Final Sample of Precipitate

Salt sample before experiment	-White -Powdery -Brittle -Opaque
$AgNO_3$ test	There was no formation of precipitate when drops of silver nitrate were added to the solution, which means the precipitation was complete.
HCl test	No turbidity was observed when Hydrochloric Acid was added, this means the washing was complete
Final precipitate	-Light purple -Brittle -Opaque

Data:

Table 2 – Mass of the Unknown Salt Sample

	Sample A (My data)	**Sample B (Partner's data)**
Starting sample mass (±0.0001g)	0g ±0.0001g	0g ±0.0001g
Ending sample (±0.0001g)	-0.1485g ±0.0001g	-0.1455g ±0.0001g
Sample mass (±0.0002g)	0.1485g ±0.0002g	0.1455g ±0.0002g

Table 3 – Crucible Mass.

	Sample A (My data)	**Sample B (Partner's data)**
Empty crucible mass (±0.0001g)	29.4316g ±0.0001g	28.3074g ±0.0001g
Dried sample and crucible mass (±0.0001g)	29.7074g ±0.0001g	28.0135g ±0.0001g
Dried sample mass (±0.0002g)	0.2758g±0.0002g	0.2939g±0.0002g

Calculations:

Volume of $AgNO_3$ needed

Sample A:

V=($\frac{mass\ of\ sample\ (0.55)}{35.5\ \frac{g}{mol}}$/0.1$\frac{mol}{L}$)

=$\frac{0.1485(0.55)}{35.5}$/0.1 = 0.023L

= (0.023L x 1000ml) + 5ml

= 23.007ml

Sample B:

V= 27.54ml

Weight of Cl^- in precipitate:

Sample A:

1.Moles Chloride from mass precipitate:

$\frac{Mass\ of\ AgCl\ precipiate}{MAgCl}$ =

$\frac{0.2758g}{143.32g/mol}$ = 0.00192 mol

2. Mass of Chloride in precipitate

(Moles of Cl^- in precipitate) (MCl) =

(0.00192 mol)(35.453g/mol)= 0.068g

3. % Chloride in original sample

$$\frac{Mass\ of\ Cl^-\ in\ precipitate}{Mass\ of\ original\ sample}(100) =$$

$$\frac{0.068g}{0.2758g}(100) = 24.68\%$$

Sample B:

Weight of Cl^- in precipitate:

1.Moles Chloride from mass precipitate:

$$\frac{Mass\ of\ AgCl\ precipiate}{MAgCl} =$$

$$\frac{0.2939g}{143.32g/mol} = 0.00205 \text{ mol}$$

2. Mass of Chloride in precipitate

(Moles of Cl^- in precipitate) (MCl) =

(0.00205 mol)(35.453g/mol)= 0.073g

3. % Chloride in original sample

$$\frac{Mass\ of\ Cl^-\ in\ precipitate}{Mass\ of\ original\ sample}(100) =$$

$\frac{0.073g}{0.2939g}(100)$= 24.84%

4. Average %Cl in original sample:

$\frac{\%Cl\ Sample\ A+\%Cl\ Sample\ B}{2}$=

$\frac{24.68\%+24.84\%}{2}$= 24.76%

Uncertainties:

Sample A:

%uncertainty (%Cl in sample) =

%uncertainty mass Cl^- in precipitate + %uncertainty mass of original sample =

$\left(\frac{0.0002g}{mass\ of\ AgCl\ precipitate} + \frac{0.0002g}{mass\ of\ original\ sample}\right)$ x 100=

$\left(\frac{0.0002g}{0.2758g} + \frac{0.0002g}{0.1485g}\right)$ x 100 = 0.21%

Sample B:

$\left(\frac{0.0002g}{mass\ of\ AgCl\ precipitate} + \frac{0.0002g}{mass\ of\ original\ sample}\right)$ x 100=

$\left(\frac{0.0002g}{0.2939g} + \frac{0.0002g}{0.1455g}\right)$ x 100 = 0.21%

Average uncertainty:

$$\frac{\%uncertainty\ Sample\ A + \%uncertainty\ Sample\ B}{2} =$$

$$\frac{0.21\% + 0.21\%}{2} = 0.21\%$$

Relative error:

$$\frac{average\ experimental\ \%Cl - true\ \%\ Cl}{true\ \%Cl}\ (100) =$$

$$\frac{24.76\% - 55.16\%}{55.16\%}(100) = -55.11\%$$

Relative spread:

$$\frac{\%Cl\ sample\ B - \%Cl\ sample\ A}{average\ \%Cl\ of\ samples}(1000) =$$

$$\frac{24.84\% - 24.68\%}{24.76\%}(1000) = 6\ ppt$$

Discussion:

There are several reasons why the experimental value was lower than expected value and the relative error was -55.11%. one of these reasons is that the photodecomposition of AgCl occurred in air., which led to chlorine gas was released into the air; therefor decreasing the mass of Cl ions in the solution. In addition to this, since some of the precipitate particles are so small, they can easily

pass through the glass filter and this leads to a lower percentage of Cl ions. Another reason for the deviation in values is that some of the precipitate may have been left as residue on the beaker walls, which again will lead to a lower concentration of Cl ions. Lastly, the amount of $AgNO_3$ might have been miscalculated and we needed to have added more AgNO3 for complete precipitation of the salt.

Conclusion:

To conclude, the sample number of our salt was given as 361. The %Cl we obtained from our experiment was an average of 24.76%, with sample A at 24.68% and sample B at 24.84%, while the accepted value was 55.16%. The % uncertainty of the %Cl for both samples was 0.21%. in terms of precision, our results turn out precise to approximately 6ppt. in terms of accuracy, the relative error was calculated to be -55.11% due to external conditions.

Bibliography:

https://opentextbc.ca/introductorychemistry/chapter/electron-transfer-ionic-bonds-2/

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