TITLE: Gravimetric Analysis Lab

CHEM 1101

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Group: Thursday AM, Table C

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Purpose:

The purpose of this experiment was to determine the chloride content in an unknown salt sample through gravimetric analysis.

Theory:

**Ag+(aq) + Cl-(aq) 🡪 AgCl(s)**

From the above equation we see that when silver ions and chloride ions in an aqueous solution are mixed together a precipitate forms of insoluble salt silver chloride. This reaction is done in the experiment in order to collect the chlorine ions which were previously dissolved from the unknown soluble salt sample we mixed into the water in the beginning of the experiment.

AgCl is very insoluble however some solubility does occur thus the amount is represented in this solubility product:

**K(sp) = [Ag+(aq)] · [Cl-(aq)] = 1.6 x 10-10**

This number tells us that the little solubility that does occur is equal to the concentrations of silver ions and chlorine ions multiplied together, which produces a very small number that is considered negligible. Since this solubility is negligible, we consider that pretty much all the chlorine ions are consumed by the reaction of the silver and chlorine ions producing silver chloride.

By precipitating in acid media, we can prevent co-precipitation from anions of weak acids, such as CO32- for instance, which occurs when rapid precipitation occurs. Additionally, by precipitating in acid media we area also able to prevent precipitate from reversing to its colloidal state.

By heating our solution, the colloids consisting of extremely small particles (which usually don’t have the time to form a solid on their own) are able to collide more frequently and thus speed up the process of making a solid. Through this process the coagulation of the precipitate takes on a more crystalline form and the precipitate is less likely to pass through the filter paper due to the precipitate’s qualities of being a denser curdy mass.

**AgCl(s) 🡪 Ag(s) + Cl2(g)**

The equation above is showing the effects of photodecomposition. This reaction happens when dry silver chloride decomposes into silver and chlorine in the presence of light. The chlorine is released as a gas and thus not contained within the precipitate anymore. This would cause our final chlorine quantity in our unknown salt to be lower than it actually is. Photodecomposition can also occur due to the presence of excess silver ions in our aqueous solution. This is illustrated in the reaction:

**3Cl2(g) + 3H2O(l) + 5Ag+(aq) 🡪 5AgCl(s) + ClO3-(aq) + 6H+(aq)**

This reaction causes our final results to have a higher chlorine quantity in our unknown salt than there actually is. This is due to the chlorine gas converting back into silver chloride instead of being released into the atmosphere.

The amount of precipitate lost by washing with 100 mL of fresh water is calculated first by knowing the concentration of [Cl-(aq)] that is lost due to solubility. This is derived from the Ksp value where:

**Ksp = X2= [Ag+(aq)] · [Cl-(aq)] = 1.6 x 10-10**

As a result, **X = [Ag+(aq)] = [Cl-(aq)] = [AgCl] = 1.26 x 10-5 mol/L**. This is the ratio for the amount of silver chloride lost. The molar mass of **AgCl = 143.32 g/mol**. And since we are using 100 mL of fresh water:

x x 0.100 L = 1.81 x 10-4 g

Thus, 1.81 x 10-4 g of AgCl is lost by washing with 100 mL of fresh water. However, this is a very small amount so it is negligible.

Some ions that may co-precipitate with chloride ions include CO32- (carbonate) since it is present in the atmosphere and Pb2+ (lead) if there is some left over from any of the beakers from a previous experiment. These scenarios as a result will increase the mass of our precipitate. And if the mass of our precipitate is increased, having precipitates that are not only silver chloride, then there will be a lower value of Cl- calculated.

Procedure:

An unknown salt sample #(insert) in a vial was taken and weighed on an analytical balance. (insert)g of the salt sample was weighed out through weighing by difference. Some of the salt was taken out and put into a 250 mL beaker and then the original mass of the salt sample is subtracted by the new mass of the vial in order to find the mass of the salt in the 250 mL beaker. 100 mL of distilled water and 1 mL of dilute HNO3 was then added to the 250 mL beaker with the salt sample. The solution was stirred until the salt sample was completely dissolved. The quantity of AgNO3 needed was calculated, which was (insert) mL, and then added to the solution with an additional 5 mL of AgNO3 so that all silver ions would react with all the chloride ions. The solution was then put on a hot plate and stirred until condensation formed around the inner walls of the beaker or almost boiling. At this point, the beaker was removed from the hot plate and a few drops of AgNO3 were added to ensure that all the chloride ions had precipitated with the silver ions. Once no more precipitate was forming, the solution was kept in a dark drawer in order to prevent the effects of photodecomposition. A crucible was then taken and weighed on the analytical balance. The crucible was then placed on the filtration apparatus and the vacuum was activated in order for the filtration process to work. The mixture was decanted until all liquid was gone. The beaker was then washed with HNO3 in order to decant the washings through the filter. The HNO3 washing was done once more in order to get the remaining precipitate out of the beaker by transferring the precipitate onto the filter. Distilled water was used to wash out any remaining precipitate in the beaker onto the filter. The washings were then collected and tested for any remaining silver ions by adding a few drops of HCl. A washing was done again, this time the precipitate was washed with three 5 mL portions of acetone. The apparatus was then disassembled and the crucible was then placed in the oven for (insert) minutes. The crucible was then cooled for (insert) minutes in a desiccator and weighed. The original mass of the crucible was then subtracted from this new mass with the precipitate in order to get the mass of the silver chloride.

Observations:

|  |  |
| --- | --- |
| **Phase** | **Observations** |
| **Phase 1 – sample** | * White colour * Powdery substance * Solid |
| **Phase 2 - solution and testing for completion** | * Solution turned white when silver chloride was added * As stirred over heat solution became clearer * Adding drops of AgNO3 caused more precipitate to form until no more precipitate was forming * No changes were observed when adding drops of HCl to the washings |
| **Phase 3 – precipitate** | * Light purple colour * Curdy-like appearance |

Data:

|  |  |  |
| --- | --- | --- |
| **Sample #358** | **Mass of Sample (g)** | **Mass of AgCl(s)** |
| Ali’s Trial | 0.1337 ± 0.0002 | 0.3063 ± 0.0002 |
| Omar’s Trial | 0.1352 ± 0.0002 | 0.2447 ± 0.0002 |

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Oven Temperature ( ֯C)** | **Time in Oven (min)** | **Cooling Time (min)** |
| Crucible | 156.5 ± 0.2 | 17 min | 5 min |

Calculations:

Ali’s Trial:

Amount of AgNO3 required:

Volume of AgNO3 needed (mL) = Mass of sample × 0.55 ÷ 35.5 ÷ 0.1 ×1000 + 5

= 0.1337 g x 0.55 ÷ 35.5 ÷ 0.1 × 1000 + 5

= (20.2 ± 0.2) mL

Calculation of % chloride:

mCl = MCl/MAgCl x mAgCl = x 0.3063 g = 0.07576 g

%Cl = mCl/msalt x 100 % = 0.07576 g/0.1337 g x 100 % = 56.66 %