TITLE: Gravimetric analysis of a chloride salt

CHEM 1101

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Purpose: The purpose of this experiment was to determine the amount of chloride in an unknown salt sample.

Theory: AgCl is very insoluble, and will precipitate out of solution when silver ions (Ag+) and chlorine ions (Cl-) interact. The original salt is very soluble, and in addition, chloride is removed by the silver precipitation, allowing for more to dissolve. AgCl does have a low Ksp (1.6\*10^-10), so some will be lost to the solution.

The precipitation needed to occur in an acidic environment, because otherwise AgOH would be formed, which would increase the final mass.

Procedure:

* 0.1112g of the unknown salt was weighed.
* A clean and dry sintered glass filter crucible was obtained.
* 100mL distilled water, the salt sample, and 1 mL of 6M HNO3 were added to a 250mL beaker. It was mixed with a glass stirring rod until the salt completely dissolved.
* The calculated amount of AgNO3 was added to the beaker, then heated.
* The beaker was cooled in a dark place.
* Transfer the supernatant to the crucible in the assembled filtration apparatus.
* HNO3 was added to wash the precipitate, then the whole mixture was added to the crucible.
* The precipitate within the crucible was repeatedly washed, until no more silver ions were present within the washings. This was verified by adding HCl to the wash, which should show no turbidity.
* The washings were emptied from the filtration apparatus, then the precipitate was rewashed with acetone 3 times.
* The precipitate and crucible were dried in a furnace for about 30 minutes.
* The crucible was cooled in a desiccator, then weighed. The final mass was recorded.

Observations:

The sample number was 342, and the sample was a white powder.

While the beaker was on the hotplate, some of the precipitate had become tinted slightly blue/purple.

The precipitate after was slightly purple lumps.

While testing for completeness, there was no additional precipitation when adding AgNO3, and no turbidity when HCl was added to the washing.

Data:

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| |  |  |  |  | | --- | --- | --- | --- | | Data |  |  | Uncertainties | | Starting mass | 13.1000 | g | ±0.0001g | | Final mass | 12.9888 | g | ±0.0001g | | Difference | 0.1112 | g | ±0.0001g | |  |  |  |  | |  |  |  |  | |  |  |  |  | | AgNO3 Required | 21.86 | mL | ±0.15mL | | AgNO3 Used\* | 22.22 | mL | ±0.15mL | |  |  |  |  | | Crucible mass before | 30.7375 | g | ±0.0001g | | Crucible mass after | 31.1220 | g | ±0.0001g | |  |  |  |  |
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| |  |  |  |  | | --- | --- | --- | --- | | Partner's Data |  |  | Uncertainties | | Starting mass | 13.2087 | g | ±0.0001g | | Final mass | 13.1003 | g | ±0.0001g | | Difference | 0.1084 | g | ±0.0001g | |  |  |  |  | |  |  |  |  | |  |  |  |  | | AgNO3 Required | 21.44 | mL | ±0.15mL | | AgNO3 Used\* | 21.81 | mL | ±0.15mL | |  |  |  |  | | Crucible mass before | 32.6819 | g | ±0.0001g | | Crucible mass after | 32.9067 | g | ±0.0001g | |  |  |  |  |

The oven was 122.1 degrees Celsius, and the crucible was in it for 30 minutes. The crucible was cooled for 10 minutes afterwards. The uncertainty for the times is 20 milliseconds.

Calculations:

See attached sheet.

Discussion:

Gravimetric analysis, by design, allows for fairly accurate determination of chlorine content within water soluble salts. Particularly, the Ksp of AgCl is very low, so only tiny amounts would have been lost in the small amounts of water used.

Factors that can increase the calculated chloride value include: other anions which could precipitate with silver, decomposition of AgCl and reformation of AgCl from chlorine gas, and insufficient washing leading to trapped mass.

Factors that can decrease the calculated chloride value include: Loss of AgCl due to washing, incomplete precipitation, and decomposition of AgCl and loss of chlorine gas.

However, the largest factor in my calculations would be an accidental spill before the acetone wash. I attempted to retrieve all the scattered precipitate and most likely picked up other material in the process. This would greatly inflate the final mass and % chlorine calculated.

This hypothesis is consistent with the calculations done with my partner’s data. His data is consistent.

Conclusion:

In this experiment, we used gravimetric analysis to determine the chlorine content of sample #342. An accidental spill caused inaccuracies in the final mass. The calculated value of chlorine in this experiment was inaccurate, at 85.53%, vs the known value of 53.83%. Using my partner’s data, the calculated value is at 51.30%, which is much closer to the known value. The discrepancy of my value and the theoretical value is therefore caused by the accidental spill.