

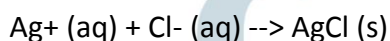
Title:

Gravimetric verification of chloride concentration from a precipitate of silver nitrate.

Introduction:

Gravimetric analysis is a method of quantitative chemical analysis used to determine the concentration of a constituent by converting it into a substance of known composition and separating it from the sample. It is then measured by weighing the change in mass from the isolated substance and the original constituent. The aim of this experiment is to verify that the concentration of chloride ions from a particular water sample is accurate, by analysing and comparing its gravimetric effectiveness through measuring the weight of its precipitate and calculating the percentage of chloride in the original sample. It is predicted that by calculating percentage concentration of chloride, the constituent will measure the same weight as the original concentration of 6.9g/L.

Precipitation is a method in which insoluble solids emerge from a liquid solution. In this particular experiment, the concentration of chloride ions is verified by adding a solution of silver nitrate (AgNO₃) to a sample of chloride ions, thereby forming a precipitate of silver chloride (AgCl). The precipitation reaction between chloride ions and silver nitrate forms the following ionic equation:



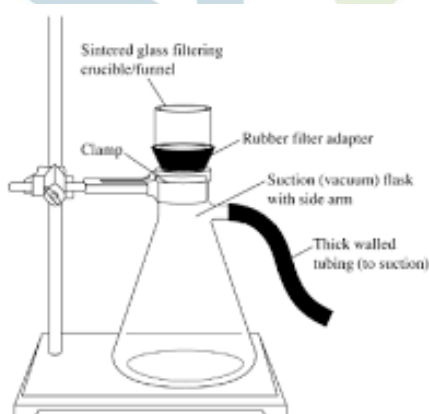
This precipitation reaction between these cations and anions results in the salt product of sodium chloride. Excess silver nitrate is added to ensure that all of the chloride ions have been precipitated fully. If not enough silver nitrate is added, the precipitation will be inaccurate, thereby resulting in a lower calculated percentage of chloride in the sample of water.

After precipitating the constituent, chloride ions are isolated from the solution by filtration and weighed. The weight of the filter paper and precipitate is used to calculate the percentage of chloride ions in the sample and verify the accuracy of the specific labelled concentration.

There are various requirements for the chloride ion concentration to be calculated accurately. Solutions that are known to have a significant concentration of chloride ions would be more beneficial in calculating a more precise verification of the required concentration. The reactant must react solely with the constituent of interest to form the correct precipitate, through minimising the variation of ions present in the sample. It must also only form one product so that it can be validly filtered and measured.

Method:

1. The brand of water was recorded and the chlorine content was stated in the results table. [**SAFETY:** *Water in the laboratory should not be consumed due to potential chemical contamination. Any water spilled on the floor must be cleaned to prevent slip hazards*]
2. A 10 ml sample of water was prepared in a 100 ml glass beaker. [**SAFETY:** *Glass beaker must be handled with care; if breakage occurs, discard the beaker and sweep up broken glass with a dustpan, do not use fingers.*]
3. 30 ml of 0.1 M Silver Nitrate was measured using a measuring cylinder. It was added to the water drop by drop using a pipette to ensure accuracy and control of precipitation reaction. The solution was continuously stirred. A precipitate of Silver Chloride was formed. To ensure that all chloride ions were precipitated, excess silver nitrate was dropped into the solution. [**SAFETY:** *Do not allow solution to enter eyes; wear safety glasses to prevent any chemicals from making contact with the eyes. Contact with silver nitrate may cause eye damage at high concentrations. It may cause temporary skin and eye irritation.*]
4. The precipitate was allowed to settle for 2 minutes.
5. A piece of filter paper was weighed on the electronic balance and recorded on the results table to 3 decimal places. [**SAFETY:** *Ensure that wiring on electronic balance is not defective to prevent danger of electrocution, particularly in wet areas near the water solution*]
6. The filter paper was placed into a Buchner funnel. The funnel was connected to the tap.



7. The solution was poured into the Buchner funnel so that precipitate was collected on the filter paper. [**SAFETY:** *After use of filter paper, dispose of its contents appropriately. Filter paper is flammable*]
8. Deionised water was used to rinse out the remaining precipitate from the 100 ml beaker into the funnel after the liquid was removed from the initial solution.
9. The precipitate collected was carefully removed along with the filter paper. It was placed on a watch glass and left in the oven to dry overnight.
10. The filter paper and precipitate were weighed on the electronic balance to 3 decimal places, before the results were recorded.
11. Steps 1-10 were repeated 3 times. [**Risk Assessment is attached**]

Results & Discussion

The labelled chloride content in a particular water sample was 6.9g/L. Through quantitatively measuring the concentration of chloride and producing a precipitate from silver nitrate, the gravimetric calculation was determined to be 6.97g/L, which was 1.014% different than the stated amount. The hypothesis that the constituent will measure the same weight as the original concentration is supported, as there is only a slight difference in concentration, which is highlighted in the evaluation section of this report. Differences in concentration may have been as a result of outlying values, which were not repeated due to time constraints.

All 4 trials generally showed consistent results averaging approximately 0.2815 grams of the resultant precipitate Silver Chloride, as shown in Table 1. When Silver Chloride (AgNO_3) is added to the solution containing Chloride ions, a displacement reaction occurs, where Silver and Chloride ions precipitate quickly due to their insolubility. This occurs until all of the chloride is consumed, because chloride is the limiting reactant in this chemical reaction.

Table 1: Measurement of Silver Chloride Precipitate through Filtration

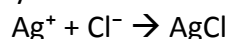
Trial Number:	Filter Paper + Precipitate (grams)	Filter Paper (grams)	Precipitate (grams)
1	0.660	0.367	0.293
2	0.609	0.329	0.280
3	0.611	0.333	0.278
4	0.646	0.371	0.275

Table 2: Mean Precipitate Measurements of Silver Chloride

Precipitates	Mean (grams)
Trials 1-4	$(0.293+0.280+0.278+0.275)/4$ $= 0.2815$
Maximum-Minimum Precipitates	$(0.293 + 0.275)/4$ $= 0.142$

Calculations:

Ionic Equation of Precipitate is given by:



Number of Moles of Silver:

$$n(\text{Ag}) = 0.2815 / (35.5 + 107.9) \\ = 0.001963 \text{ mol}$$

Mole Ratio:

Ag^+ and Cl^- are balanced in this ionic equation
Therefore, the mole ratio is 1:1

Number of Moles of Chloride:

$$n(\text{Cl}) = 0.001963 \text{ mol}$$

Mass of Chlorine in each sample:

$$\begin{aligned} & 0.001963 \times 35.5 \\ & = 0.069687936 \\ & = 0.006969 \text{ per } 10\text{mL} \\ & = 6.97 \text{ g/L} \end{aligned}$$

Percentage Error:

$$\begin{aligned} & (6.97-6.9)/6.9 \times 100 \\ & = 1.014\% \end{aligned}$$

Graph 1:

Discussion:

Gravimetric analysis involves calculating the concentration of a substance of unknown composition from a substance of known composition. By reacting chloride to form a precipitate of known amounts and isolating the precipitate from filter paper, the final weighed product determines the concentration of the substance of previously unknown composition. When the Silver Chloride precipitate formed, a white cloudy substance was formed and coagulated on the bottom of the beaker after 2 minutes of letting the solution settle.

The precipitation of Silver Chloride is formed through solubility rules. Salts such as Cl^- , Br^- and I^- are mostly soluble. Insoluble substances include Ag^+ and Pb^{2+} . Thus Silver Chloride is an insoluble substance, and only substances of these requirements are able to form a precipitate. When silver nitrate was added to the chloride solution, a double displacement reaction occurred. It was observed that the solution became white and milky, thereby demonstrating that silver chloride was formed. After drying and measuring the precipitate, the average weight of solid precipitation was 0.2815 grams.

The purpose of this experiment was to ensure that the chlorine content in water was the same as the stated figure through a method called stoichiometry. The theoretical yield of chlorine concentration in the labelled water is expected to be 6.9g/L. However, after conducting the experiment, it was found that the actual yield of chlorine ions was approximately 6.97g/L. The percent yield of chloride was calculated to be 1.014% different to the labelled figure. This small difference was demonstrated to be due to other experimental and method-related errors that occurred unknowingly throughout the experiment. Such limitations, if avoided, would result in a more accurate figure (closer to 6.9g/L. All 4 trials were relatively consistent. However, the result from Trial 1 of Table 1 is much higher than trials 2-4, consequently resulting in an increase in the final 6.9g/L figure to 6.97g/L. These limitations will be further discussed in the *Evaluation* section.

Evaluation:

The experiment overall yielded a higher result than expected, with a concentration of 6.97g/L rather than the expected concentration of 6.9g/L. The 1.014% error (as shown in the calculations section of the practical report) may be due to too much excess Silver ions when testing for the complete formation of the precipitate with present ions other than chloride.

Therefore, when verifying the sample, the average precipitate figure would have varied and resulted in a higher/lower measurement. For future experiments, the volume of precipitating agent should be calculated and measured with a burette to increase the accuracy of adding an appropriate amount of silver nitrate into the solution.

$$\begin{aligned}n &= m/M \\n &= 6.9/35.5 \\&= 0.00194 \text{ mol}\end{aligned}$$

Volume of precipitating agent:

$$\begin{aligned}V &= 0.00194/0.1 \\&= 0.0194 \times 1000 \\&= 19.4\text{mL to be measured with a burette}\end{aligned}$$

By adding 19.4mL of silver nitrate into the solution, the remaining excess silver nitrate can be dropped mL by mL. This method should be taken in intervals of 1 minute to qualitatively determine if more silver nitrate should be added into the solution to complete the precipitation process.

When the precipitate was washed down with deionised water into the Buchner funnel, the water could have been mistakenly contaminated with chemicals due to the nature of the chemical laboratory and exposure to various substances. Furthermore, improper washing of the precipitate may have brought in unwanted ions to the precipitate, thereby affecting the final concentration of each precipitate. For example, Trial 1 had a precipitate weighing 0.293, whereas Trial 2 had a precipitate weighing 0.280. These results are significantly different, and may possibly be due to the suggestions made previously. The inconsistency in the final precipitate masses follows the same trend in the Filter + Precipitate column of Table 1. Trial 1 measured 0.660 grams, whereas Trial 2 measured 0.609 grams, showing the same significant fluctuation in results.

The differences in filter paper weight may be as a result of the scale not being properly calibrated. It could also be caused by using two different sets of electronic balances, both of which have different levels of calibration. For example, 4 pieces of filter paper from the same brand were weighed out to be 0.367, compared to 0.329 for Trial 2 filter paper, and 0.371 for Trial 4. This caused a slight difference in weighing the filter paper, thereby decreasing accuracy in calculation of unknown concentration throughout the experiment. For future experiments, the filter paper should be weighed one at a time, on the same electronic balance after calibrating and taring it to ensure maximum validity of results.

Conclusion:

Unknown concentrations of chloride were analysed through gravimetric determination to verify their molecular accuracy. The 4 trials contained 6.97g/L concentration of chloride, compared to the original sample which contained 6.9g/L concentration; a 1.014% difference was produced due to systematic and human errors, for which suggestions have been made to confer these errors in future experimentation. The main finding of the experiment was

that by isolating the chloride from a precipitate, the concentration of chloride ions could be accurately measured through gravimetric analysis. It was found by adding silver nitrate into the water solution containing chloride. Due to the solubility of nitrate, it was dissolved, and the insoluble substances (chloride and silver) were reacted to form silver chloride. This was precipitated out of the solution and isolated by filtering the remaining precipitate solids. Hence, the remaining measured content was able to be calculated. Overall, the calculated concentration was acceptable as only minor errors would have been made to create an increase in concentration of chloride ions in the water sample. Aside from these factors, the hypothesis that by calculating the concentration, the constituent would measure approximately the same weight as the original concentration was supported. It was also supported that the method of gravimetric analysis was an effective in determining quantitative data for unknown samples of ions, yielding relatively accurate results compared to other methods of experimentation.

