



# Gravimetric Analysis

Experiment  
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*Quantitative analysis* is a method used to determine exact amounts or concentrations of an unknown *analyte*. An analyte is a substance that is analyzed by some scientific procedure. *Gravimetric analysis* is a method in quantitative analysis where an unknown sample is dissolved in an appropriate solvent, and the analyte is converted to an insoluble form of a known compound. The precipitate is then collected, dried and weighed. From this weight, the amount or percent of the analyte in the original sample can be determined.

In this experiment the chlorine content of an unknown soluble chloride salt is to be determined. The unknown is dissolved in distilled water and dilute silver nitrate solution is added to precipitate out the chloride as silver chloride (AgCl).

The percent of chloride in the unknown is calculated as follows:

$$\% Cl = \frac{\text{mass of Cl in sample}}{\text{mass of sample}} \times 100$$

The mass of Cl can be determined from the fact that it is precipitated and weighed as insoluble AgCl. The % composition of Cl in AgCl can be calculated as follows:

$$\text{mass of Cl in sample} = \text{mass of AgCl} \times \frac{\text{Atomic weight of Cl}}{\text{Molecular weight of AgCl}}$$

Thus, by substitution:

$$\% Cl = \frac{\text{mass of AgCl in sample}}{\text{mass of sample}} \times \frac{\text{Atomic weight of Cl}}{\text{Molecular weight of AgCl}} \times 100$$

The ratio of the atomic wt of Cl to the molecular weight of AgCl as determined by % composition method is called GRAVIMETRIC FACTOR. For example if the chloride were precipitated as PbCl<sub>2</sub> then the gravimetric factor would be

$$\frac{2 \times \text{Atomic Weight of Cl}}{\text{Molecular weight of PbCl}_2}$$

## EQUIPMENT

50 mL, and 400 mL or 500 mL beakers; Buchner Funnel; vacuum flask, filter paper; glass stirrer (with or without a policeman); iron ring, wire gauze, and aluminum foil.

## PROCEDURE

(Day 1)

### Part I – PRECIPITATION OF THE CHLORIDE

1. USE EYE PROTECTION
2. YOU MUST USE DISTILLED WATER. All glassware must be rinsed with distilled water.
3. Obtain an unknown chloride sample and weigh accurately between 0.2 to 0.3 grams of the sample into a pre-weighed 50 mL beaker.
4. Carefully transfer this to a 400ml or a 500ml beaker. Again carefully wash your weighing bottle into the beaker. Add approximately 150 mL of distilled water and stir to dissolve the sample.
5. Add approximately 1ml or 1:1 nitric acid (or 0.5 mL of concentrated nitric acid) to the solution in the beaker.
6. Use a glass dropper and slowly add 0.1 M (or 0.2 M) silver nitrate solution with stirring until no precipitate is observed (avoid excess silver nitrate). Place your beaker on the flame and heat to nearly to boiling while continuously stirring the solution. *You may have to perform this procedure in subdued light.*
7. Do not heat for more than 2 minutes after it reaches boiling. The precipitate will coagulate. Remove the beaker and allow the precipitate to settle. Test for complete precipitation by adding a few more drops of silver nitrate. A white cloudiness at this point means that you should add more silver nitrate, you should stir and heat the solution as you did before. (AVOID EXCESS  $\text{AgNO}_3$ ).
8. Cover the beaker, wrap it in foil and let it stand for two hours or until the following laboratory period. (The reaction is light sensitive so store the beaker in a dark cabinet or locker).

### DATA FOR PART 1

Unknown Number	
Mass of empty weighing bottle	
Mass of bottle plus sample	
Mass of sample	

(Day 2)

Part II – FILTRATION AND WEIGHING OF THE PRECIPITATE

1. Weigh your filter paper on the analytical balance.
2. Place the weighed filter paper in the funnel mounted on the filtering flask attached to a vacuum system. Wet the filter paper with DI water and turn on the vacuum system so that there is a gently suction through the system.
3. Decant your solution through the crucible by pouring the solution down a stirring rod. Make sure that the precipitate is disturbed very little.
4. Wash your precipitate in the beaker by adding approximately 25 mL of 0.01M nitric acid. Stir the solution, let the precipitate settle down and then decant again through the crucible. Repeat this washing two more times.
5. REPLACE THE WATER in the wash bottle with the 0.01 M nitric acid solution.
6. Transfer the precipitate to the crucible. Use a glass rod with or without a policeman and the jet from the wash bottle. Remove any solid particles still left in the beaker with the rubber policeman. Finally use the jet from the wash bottle to wash all particles from the rubber policeman to the crucible.
7. Wash the precipitate in the crucible several times with your dilute nitric acid (you may have to test the filtrate for the presence of silver ions).
8. Remove the funnel with filter paper and product from the vacuum system and place it in a beaker and let stand until the next lab period.

(Day 3) – *(This will be performed with day 1 of experiment 3.)*

9. Weigh a clean dry watch glass.
10. Remove the dry filter paper from the funnel, place it on the pre-weighed watch glass and then weigh both on the analytical balance.
11. Clean the Buchner funnel and vacuum flask.

DATA FOR PART II

Unknown Number	
Mass of unknown sample from Part I	
Mass of empty filter paper	
Mass of watch glass	
Mass of filter paper + watch glass + AgCl	
Mass of AgCl	
Gravimetric factor	
% of chlorine in sample	