Analytical Chemistry

Is a branch of chemistry which is concerned with identifying the constituents of the chemical and the possibility of quantification or qualitative.

Analytical chemistry is divided into two branches:

1. Qualitative analytical chemistry:

It is concerned with the knowledge of constituent elements types of the compound It also looks at how the elements or materials are separated and identified and is concerned with the external appearance of the compound such as color and smell.

2- Quantitative Analytical Chemistry:

It is concerned with quantifying the elements or compounds in a sample. Which in turn are classified into three parts:

A. Volumetric analysis:

It is one of the ways by which the amount of elements or compounds in a sample solution is estimated .

1- Precipitation method.

2- Volatilization method.

3- Specific gravimetric methods.

4-Methods of electrostatic deposition Electro gravimetric methods

B. gravimetric analysis:

It is based on the estimate of the material by weighing it or by any other method. The material is estimated as a weight in grams or its parts or percentage in a particular model and is divided into:

C. Instrumental analysis:

we prefer the methods of volumetric analysis more than the methods of gravimetric analysis, knowing that the latter is more accurate than the previous one this is due to the slow of the gravimetric analysis.

The precipitation method is the best method used in gravimetric analysis.

The sample is transferred to the solution to be analysed in a suitable manner. The element to be evaluated is precipitated as a non-dissoluble compound, after which the precipitate is filtered, washed, ignited or dried.

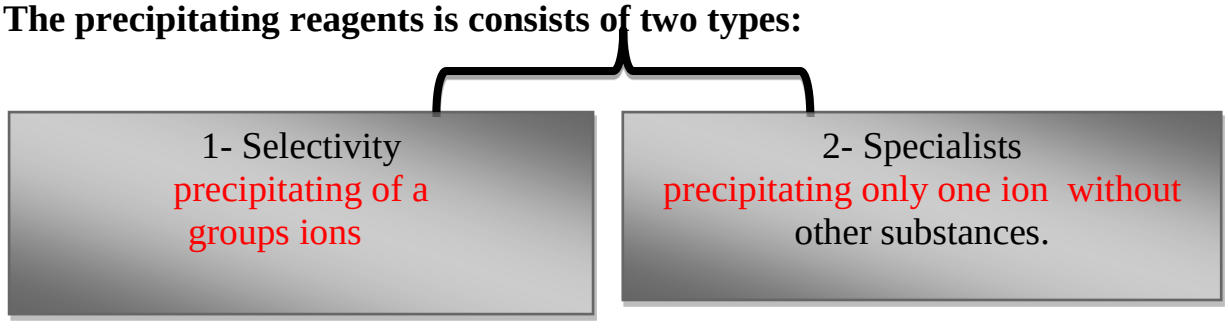
Properties of precipitates in the methods of gravimetric analysis:

1- Must be known chemical formula.
2- The solubility of precipitate is very low.
3- The crystals of the precipitate of an appropriate size (large).
4- The precipitate should be not contain contaminants and can removedit be washed and drying the precipitate.
5- The precipitate will remain stable at the drying temperature (thermally stayble).
6- The Molecular weight of the precipitate is large compared with the material to be estimated to avoid error.

The precipitating Reagents

It is the chemical reagent that causes the precipitation of the soluble analyle to be estimated, forming precipitate or low soluble salts in the water .

The precipitating reagents is consists of two types:



How to per form asuccessful gravimetric analysis?

1- Preparation of the solution.
2- Precipitation.
3- Digestion.
4- Filtration.
5- Washing.
6- Drying or igniting.
7- Weighing and calculation.

1- Preparation of solution

This is done by selecting a suitable weight from the sample to be estimated and then dissolving the sample by selecting the appropriate solvent.

As well as to create a suitable conditions for the experiment, such as pH of solution and solution temperature.

2- Precipitation:

The precipitation process of the selected components from the rest of the components in the solution take place or occure by adding the precipitating reagent.

The precipitate ion process consists of two steps :

First : The formation of small particles (1-100) nm, called nuclei.

Second: The nuclei growth into large particles. The nuclei do not appear directly after the addition of the precipitating agent but often is noticed after a period called the induction period. The duration of the sedimentation varies with the sediment difference and ranges from the second parts of silver chloride to a few minutes in the case of barium sulphate.

3. Digestion of precipitation:

It is the process of heating the precipitate with the mother liqud (usually 90 or 95C$^{\circ}$ and leaving it a period of time.

Advantages of digestion:

1- Increased the solubility of small particles (Help the crystals growth).
2- Leave the precipitate with the mother liquor for a period of time allowing these particles to be precipitate on the large particles.
3- helps the grouping of colloidal precipitate.

4 – Filtration:

The purpose of the filtration process is to separate the precipitate from the mother liquor the filtration process is easier and faster if the precipitation crystals are large while this process becomes more difficult if the crystals are small.

It is used for this purpose:

1- Ashless filter paper
2- the base of asbestos furnished on the inner bottom of the crucible.
3- crucible have porosity for filtration.

The fast filtration is best to prevent post precipitation, which considers one of the types of contaminants.

5- Washing:

The impurities,especially those on the surface, can be removed by washing the precipitate after filtering. The precipitate will be wet with mother liquor, which is also removed by washing.

6- Drying and igniting:

The precipitate must be heated to remove water and to remove the adsorbed electrolyte from the wash liquid.This drying can usually be done by heating at 110 - 120C°. Igniting at a much higher temperature is 250 C° usually required if a precipitate must be converted to a more suitable form for weighing.

7- Weight:

After finishing the process of igniting or drying the precipitate, transfer the precipitate to the desiccator and leave it in the desiccator until it is cooled at room temperature.

General rules for obtaining a precipitate with large crystals:

1- precipitation from diluted solutions.

2- Make the acidic function of the solution suitable for the precipitation process because some materials prefer to be precipitate in the acidic media, while other materials prefer precipitation in the basic media.

3- Slow addition with continuous stirring prevents local excesses of the reagent .

4- In the precipitation of hydroxides, it is preferable to use buffer solutions for the purpose of avoiding the development of high local PH centers and this can be avoided by precipitation from homogeneous solutions.

5- It is preferable to digest the precipitate if there is no risk of post- precipitation.

6- Wash the precipitate with a suitable solution do not react with the precipitate and do not increase its solubility and prefer to wash the precipitant on several small batches.

precipitation from Homogenous Solutions :

Is one of the precipitation methods in which the precipitation agent is composed slowly, uniformly and homogeneously in the solution either through the hydrolysis of a substance or the interaction of two substances with each other such as:

A- Oxalates are generated from the hydrolysis of dimethyl or diethyl oxalate, which is used as a precipitating agent for calcium.

$$(C_2H_5)_2C_2O_4 + 2H_2O \rightleftharpoons 2C_2H_5OH + 2H^+ + 2C_2O_4^{=}$$

B- **Sulfates where sulfate ion can be generated from hydrolysis of sulfuric acid.**

$$NH_2SO_3H + H_2O \rightleftharpoons NH_4^+ + H^+ + SO_4^{-2}$$

Advantages of precipitation of a homogeneous solution :

1- **The crystals are slowly precipitated large, regular and easily filtered.**
2- **The formed Precipitate is thermally stable and is easy to dry.**
3- **The precipitate will be free from impurities.**

Disadvantages of precipitation of a homogeneous solution :

1- **Precipitation requires a homogeneous solution for a long time compared to normal Precipitation methods.**

2- **Ahigh-cost method because Precipitators and solvents are used compared to what method normal Precipitation.**

Impurities in precipitates:

precipitate tend to carry down form the solution other constituent that are normally soluble, causing the precipitate to become contaminated.

It gets in two cases:

1- Co precipitation

Includes all Contaminants obtained during the composition of the precipitation required to be analyzed and includes:

- **A- True precipitation.**
- **B- Occlusion and inclusion.**
- **C- Surface adsorption.**

2- post precipitation

Which is the Contamination of the precipitation after its formation. The precipitate is composed of pure and solid crystals during precipitation and then it is contaminated by precipitator of another substance. This type of contamination can be eliminated by filtration immediately after precipitation.

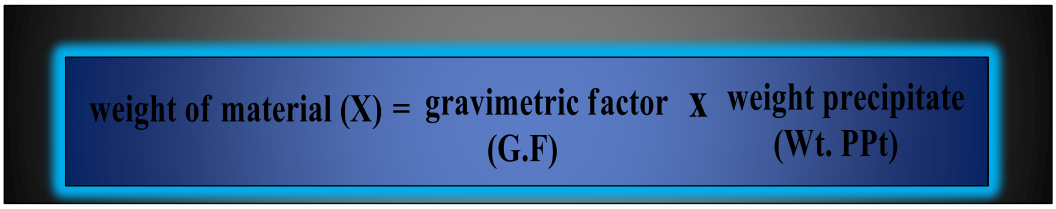
Calculation

The calculations for gravimetric analysis Depends on two things:

1- **The sample.**
2- **Material to be estimated.**

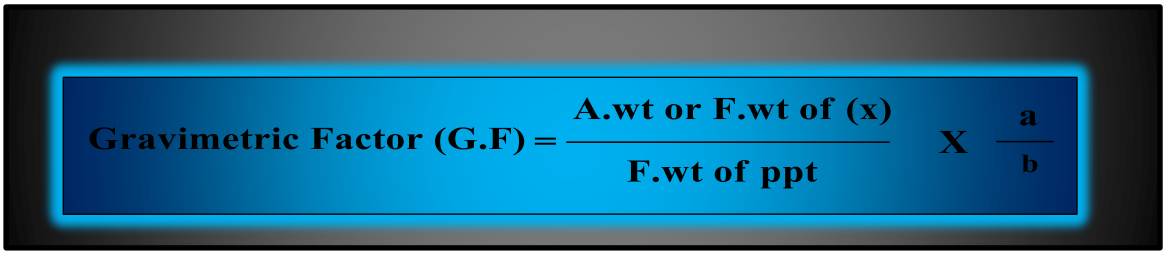
The two are then linked to find the percentage and concentration of the material required.

$$\text{Percentage of the material X(\%)} = \frac{\text{weight of material}}{\text{weight of the sample}} \times 100$$



Gravimetric Factor (G.F)

Is the weight of the formula or the atomic weight of the material to be analyzed to the weight of the formula of the known weight precipitate and does not depend on the weight of the original form which is a fixed quantity.



Note: If there is a common atom in the ridge and the place (except oxygen), we must multiply the numerator or the place or both with a number or two different numbers so that the number of this common atom is equal in the numerator and denominator.