CHAPTER – 4 Gravimetric Analysis

4.1 INTRODUCTION

Gravimetric analysis is a technique through which the amount of an analyte (the ion being analysed) can be determined through the measurement of mass.

4.2 PRINCIPLE OF GRAVIMETRIC ANALYSIS

The principle behind the gravimetric analysis is that the mass of an ion in a pure compound can be determined. Later, used to find the mass percent of the same ion in a known quantity of an impure compound. It is a quantitative analysis method by weight.

The four main types methods involved in the gravimetric analysis:

1. Precipitation method
2. Volatilization method
3. Electro-analytical method
4. Miscellaneous physical method

4.3 TYPES OF GRAVIMETRIC ANALYSIS

There are four fundamental types of gravimetric analysis.

1. Volatilization gravimetry: Volatilization Gravimetry involves separating components of our mixture by heating or chemically decomposing the sample.

2. Precipitation gravimetry: Precipitation Gravimetry uses a precipitation reaction to separate one or more parts of a solution by incorporating it into a solid.

3. Electrogravimetry: Electrogravimetry is a method used to separate and quantify ions of a substance, usually a metal.

4. Thermogravimetric: Thermogravimetric is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature or as a function of time.

4.4 STEPS IN A GRAVIMETRIC ANALYSIS

Steps in a gravimetric analysis After appropriate dissolution of the sample the following steps should be followed for successful gravimetric procedure:

1. Preparation of the Solution: This may involve several steps including adjustment of the pH of the solution in order for the precipitate to occur quantitatively and get a precipitate of desired properties, removing interferences, adjusting the volume of the sample to suit the amount of precipitating agent to be added.

2. Precipitation: This requires addition of a precipitating agent solution to the sample solution. Upon addition of the first drops of the precipitating agent, supersaturation occurs, then nucleation starts to occur where every few molecules of precipitate aggregate together forming a nucleus. At this point, addition of extra precipitating agent will either form new nuclei or will build up on existing nuclei to give a precipitate.

This can be predicted by *Von Weimarn ratio* where, according to this relation the particle size is inversely proportional to a quantity called the relative supersaturation.

Relative supersaturation = (Q – S)/S

where

Q is the concentration of reactants before precipitation,

S is the solubility of precipitate in the medium from which it is being precipitated. Therefore, to get particle growth instead of further nucleation we must make the relative supersaturation ratio as small as possible. The condition needs to be adjusted such that Q will be as low as possible & S will be relatively large.

3. Digestion of the precipitate:

- The precipitate is left hot (below boiling) for 30 min to one hour for the particles to be digested.
- Digestion involves dissolution of small particles and reprecipitation on larger ones resulting in particle growth and better precipitate characteristics. This process is called Ostwald ripening.
- An important advantage of digestion is observed for colloidal precipitates where large amounts of adsorbed ions cover the huge area of the precipitate.
- Digestion forces the small colloidal particles to agglomerate which decreases their surface area and thus adsorption.

4. Washing and Filtering the Precipitate:

- It is crucial to wash the precipitate thoroughly to remove all adsorbed species.
- One should be careful not to use too much water since part of the precipitate may be lost.
- in case of colloidal precipitates, we should not use water as a washing solution since peptization would occur.
- In such situations dilute nitric acid, ammonium nitrate, or dilute acetic acid may be used.

5. Drying and Ignition:

- The purpose of drying (heating at about 120-150^{o}C in an oven) or ignition in a muffle furnace at temperatures ranging from 600-1200^{o}C is to get a material with exactly known chemical structure so that the amount of analyte can be accurately determined.
- The ppt is converted to more chemically stable form.

6. Weighing the ppt:

- The ppt cannot be weighed accurately if placed on filter paper, nor can the ppt be completely removed from filter paper in order to weigh it.

4.5 ADVANTAGES OF THE GRAVIMETRIC ANALYSIS

1. It is precise and accurate when using modern analytical balance.
2. Gravimetric analysis can also be used to determine the atomic masses of many elements up to the extent of six-figure accuracy.
3. Gravimetry provides only very little room for instrumental error and it also does not require a series of standards for calculating the unknown.

4.6 DISADVANTAGES OF THE GRAVIMETRIC ANALYSIS

1. The gravimetric analysis, in general, can provide analysis of a single element, or a limited group of elements, at a time.
2. The chief disadvantage of this method is that it is very time- consuming.
3. The chemist in today's world prefers other methods over this method.
4. minor misstep in a procedure can often mean tragedy for the analysis.