Lec 7 Gravimetric Methods of Analysis

Dr. Rusul H. Hamza



Gravimetric methods

Selective precipitation of the analyte and non-selective measurement of mass of the precipitate (classical method)

Steps in Gravimetric analysis

- Solution Preparation (volume, pH, concentration)
- Precipitation (Formation mechanism, Crystal Size)
- Digestion of Precipitate
- > Filtration
- Washing
- Drying/Ignition
- **≻**Calculations

1. Solution Preparation (volume, pH, concentration)

hydorxyquinoline (oxine) can be made selective by pH Adjustment

Aluminium ions can be easily precipitated at pH 4.

9-hydroxyquinoline +
$$Mg^{+2}/Al^{+3}$$
 \longrightarrow $Alq_3 + Mg^{2+}$ (free)

2. Precipitation

Precipitate Process It requires addition of a **precipitating agent** solution to the sample solution

Supersaturation: The solutions contains more soluble substances than what exists under equilibrium conditions.

Nucleation: Few particle/molecules come together to form nuclei (microscopic clusters of atoms or ion) of solid phase.

Growth: The nuclei then grow by addition of other precipitate particles and form a certain shape particles.

Desired Properties of Precipitate

- -Insoluble
- Physical Form such that readily separated, washed, free of impurity
- Can be converted to Pure substance of definite chemical composition

3. Digestion of the precipitate:

Digestion involves dissolution of small particles and re-precipitation on larger ones resulting in particle growth and better precipitate characteristics

4. Washing and Filtering the Precipitate

Wash the precipitate thoroughly to remove all adsorbed species that would add to the weight of the precipitate.

5. Drying and Ignition:

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

Spectroscopy

is the study of the interaction between electromagnetic radiation and matter. The matter can be atoms, molecules or ions.

Spectrometer is something which can be used to measure the presence of particular compound or particle in a molecule

Types of spectroscopy

- 1.absorption spectroscopy
- 2.scattering spectroscopy
- 3.emission spectroscopy

Beer's law:

It stats that the intensity of transmitted monochromatic light decreases exponentially as the **concentration of the absorbing substance increases**

Spectrophotometer

that measures the amount of light absorbed by a sample.

used to measure the concentration of solutes in solution by measuring the amount of the light that is absorbed by the solution in a cuvette placed in the spectrophotometer