Gravimetric Analysis

Presentation by,

Mr. Prashant B. Patil

Assistant Professor

H R Patel Institute of Pharmaceutical Education and Research, Shirpur

Introduction

- Gravimetric analysis is concerned with the process of producing and weighing a compound or element in as pure a form, as possible some form of chemical treatment has been carried out on the substance to be examined.
- The mass of the element in the original can be then readily calculated from a knowledge of the formula of the compound and the relative atomic masses of the constituents elements.
- **Gravimetric analysis** is a technique through which the amount of an analyte (the ion being **analyzed**) can be determined through the measurement of mass.

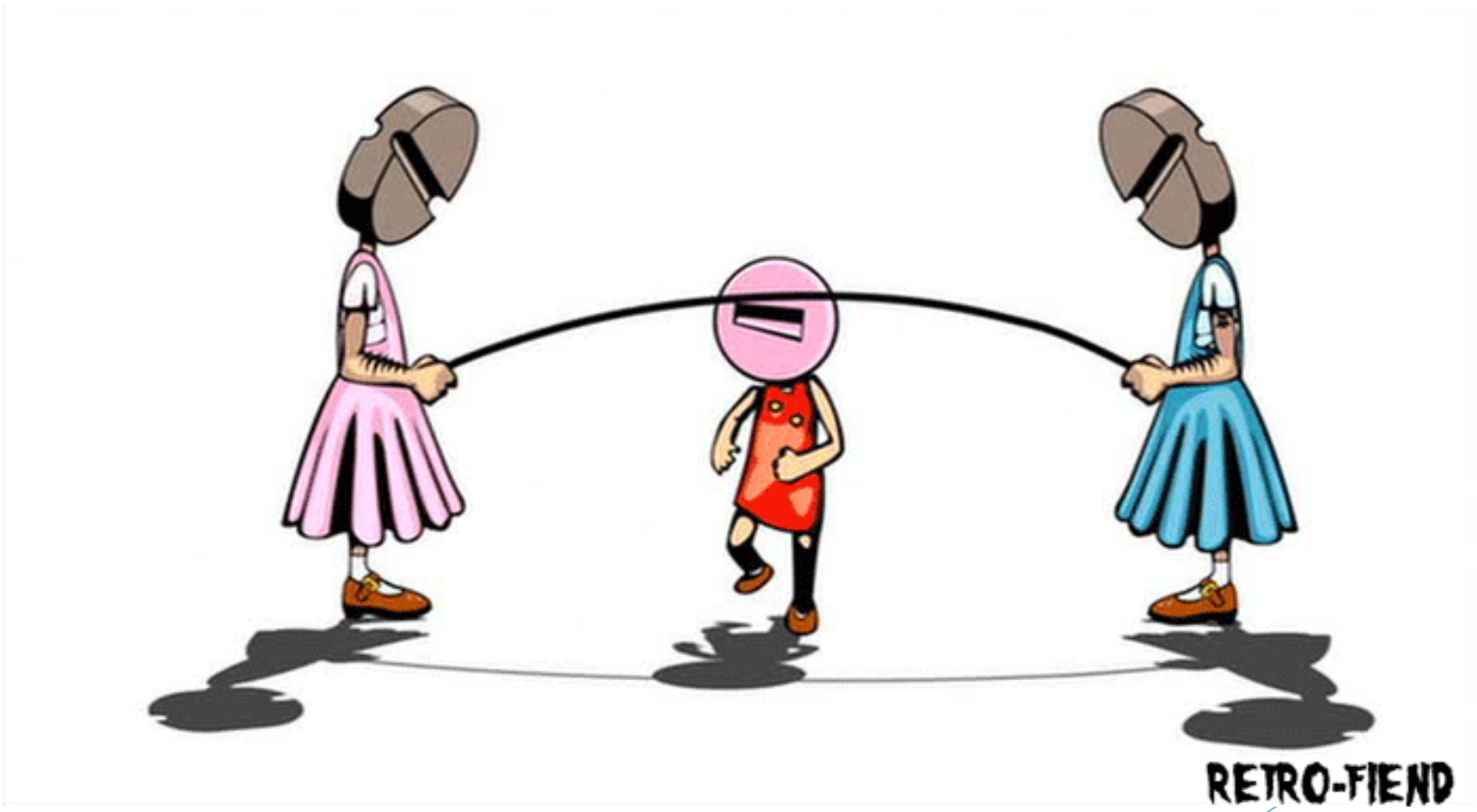
- **Advantages:**

1. It is accurate and precise
2. Possible sources of error are readily checked, since filtrates can be tested for completeness of precipitation and precipitates may be examined for the presence of impurities.
3. It is a absolute method.

- **Principle**
- The substance to be determined by converting in to pure chemical compound then weighed.

$$\%\ \text{analyte (element/radical)} = \frac{\text{Weight of precipitate} \times \text{Gravimetric factor}}{\text{Weight of Sample}} \times 100$$

$$\%\ \text{Yield} = \frac{\text{Practical Yield}}{\text{Theoratical Yield}}$$



- The separation of the element or of the compound containing element may be possible by number of ways…..
- **Precipitation Method**
- **Volatilization or evolution method**
- **Miscellaneous physical method**
- **Electro-analytical methods**

- **Precipitation –**
- **Precipitation method involves conversion of the analyte to sparingly soluble precipitate, which is then filtered, washed for free from impurities and converted to a product of known composition by suitable heat treatment. The product is weighed.**
- **Thus in determination of silver, a solution of substance is treated with an excess of sodium chloride or potassium chloride solution, the precipitated is filtered off, washed well to remove soluble salts, dried at 130-150^{o}C, and weighed as silver chloride**
- $AgNo_3^+ + NaCl^- \rightarrow AgCl$

- **The sources of contamination of precipitates affecting the gravimetric analysis include……**
- **Post precipitation**
- **Co-precipitation**

- **Post Precipitation**
- Sometimes when the precipitate is allowed to stand in presence of mother liquor the second substance will form the precipitate with reagent is called post precipitation.
- Post precipitation involves the deposition of sparingly soluble impurities of similar properties to the analyte precipitate on the surface of the latter after its formation.
- **Example:**
- Magnesium oxalate is depositing on the calcium oxalate precipitate. Calcium oxalate precipitates out in the presence of magnesium ions satisfactory without any interference but if the precipitates remains in contact with the mother liquor containing magnesium ions for a prolonged period magnesium oxalate precipitates on top of calcium oxalate.

- **Co-precipitation**
- Co-precipitation involves of the soluble substance in the precipitate during its formation.
- **Example:** to a mixture of barium chloride and potassium permanganate solution, excess of sulphuric acid is added slowly in small portions and then potassium permanganate is reduced by addition of reducing agent. After the reaction, the solution becomes colorless but the precipitate appears to be violet in color. This indicates that some potassium permanganate is co-precipitated together with barium sulphate.
- **Types of Co-precipitation:**
- **1. Surface adsorption** **2. Mixed crystal formation**
- **3. Occlusion** **4. Mechanical entrapment**

- **Surface adsorption**:
  - Adsorption depends on the surface area of the precipitate particles.
  - Colloidal particles with their large surface area enable to adsorption of different types of ions / impurities in the primary layer.
- **Mixed crystal Contamination:**
  - Mixed crystal contamination of the precipitates occurs due to substitution in the precipitate lattice with impurity ions of similar crystallinity.
- **Occlusion:**
  - Occurs during formation of precipitate when foreign ions in the counter ion layer get trapped within the rapidly growing crystal.

Steps Involved in Gravimetric Analysis

- The operation of gravimetric analysis consist of a number of important techniques to get accurate, quantitative estimation……..

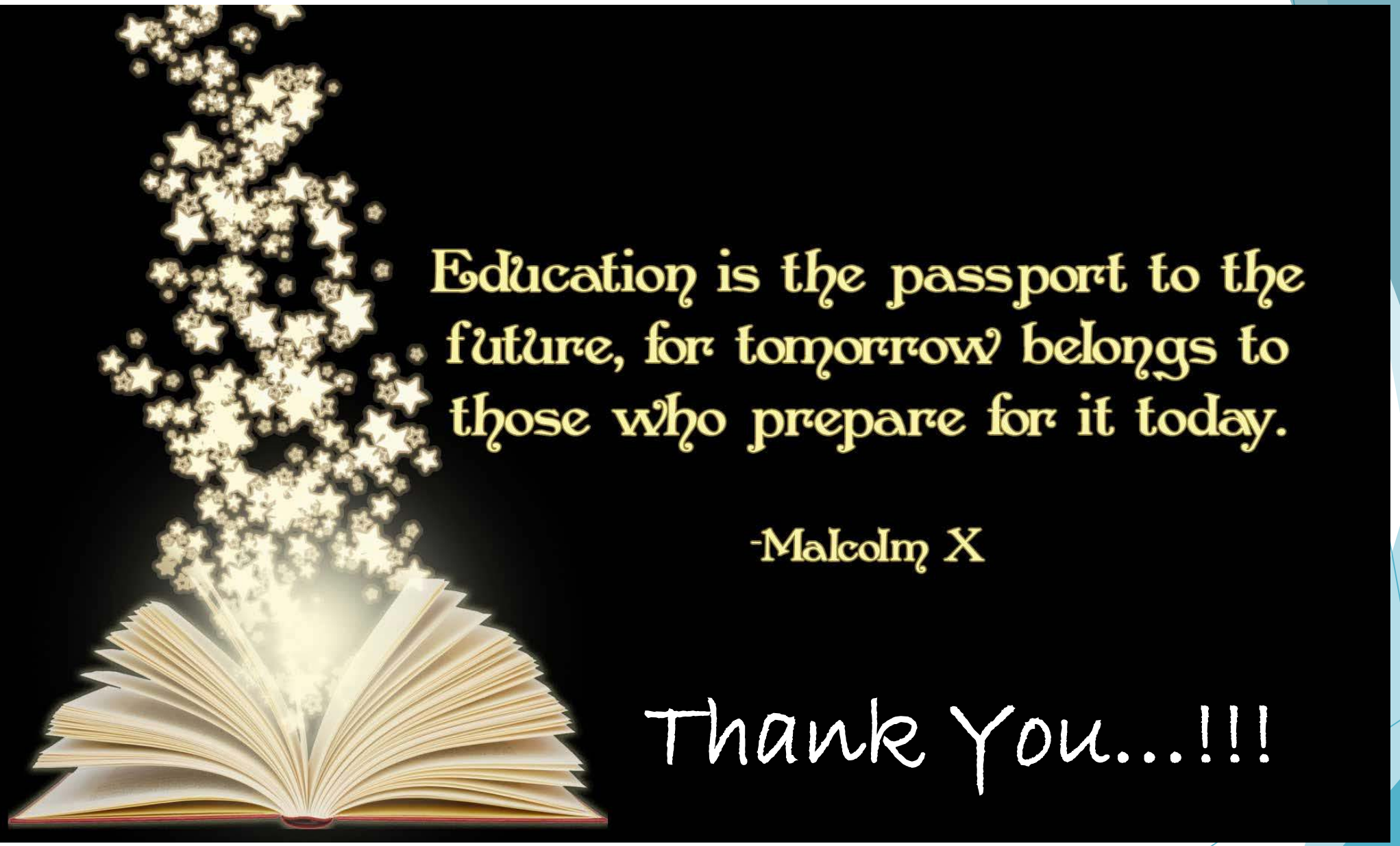
1. Sampling
2. Preparation of solution or dissolution
3. Precipitation
4. Testing the completeness of precipitation
5. Digestion or ageing of precipitation
6. Filtration
7. Washing of precipitate
8. Drying or ignition of precipitate
9. Weighing
10. Calculations

Assay of Barium Sulphate

- **Principle:** The amount of sulphate is determined quantitatively as barium sulphate, BaSO4 by gravimetric analysis. This determination consists of slowly adding a dilute solution of barium chloride to a hot solution of the sulphate slightly acidified with concentrated HCl. The white precipitate is filtered off, washed with water, dried in oven, and weighed as barium sulphate. The percentage of sulphate is calculated from the weight of barium sulphate.

- **Reaction:** $Ba_2 + SO4^{2-} \longrightarrow BaSO4$


THE GRAVIMETRIC
ANALYSIS OF BARIUM
CHLORIDE HYDRATE


Education is the passport to the future, for tomorrow belongs to those who prepare for it today.
-Malcolm X
Thank You...!!!