

**SHREE H. N. SHUKLA INSTITUTE OF  
PHARMACEUTICAL EDUCATION AND  
RESEARCH**



**B.PHARM**

**(SEMESTER -I)**

**SUBJECT NAME: PHARMACEUTICAL ANALYSIS -I**

**SUBJECT CODE: BP102TP**

**UNIT 3(c): GRAVIMETRY**

## Content

**Gravimetry:** Principle and steps involved in gravimetric analysis. Purity of the precipitate: co-precipitation and post precipitation, Estimation of barium sulphate.

### INTRODUCTION

Gravimetric analysis is the process of isolating the element or definite compound of element, in a pure form and weighing that element.

Gravimetric analysis is one of the most accurate and precise method used for large quantity.

In isolation process analyte is converted into insoluble form i.e. precipitation. The content of that analyte is then calculated from the weight of the resulting precipitates.

The isolation of component is possible by number of ways.

- (a) Precipitation method
- (b) Volatilization or evolution method
- (c) Electro-analytical method
- (d) Miscellaneous method

**A. Precipitation method** involves conversion of analyte into sparingly soluble precipitates. These precipitates then washed and make them free of impurities. By suitable heat treatment precipitates are converted to a product of known composition. Then product is weighed.

**B. In volatilization-** the analyte or its decomposition products volatilized at suitable temperature. This product is collected in form of gas and weighed. The loss of sample solution is measured which gives concentration of analyte.

**C. In electro analysis-** Element to be estimated (analyte) which is deposited electrically on a suitable electrode. Generally this method is based on Faraday's second law.

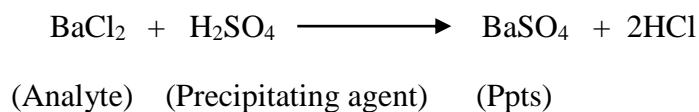
When given current is passed in series through the solution containing various ions, the amount of the substance that is deposited is proportional to current passed and in the ratio of their chemical equivalent.

Generally sample is deposited on electrode, which is separated from it by an electrical current, sample weighed and concentration of analyte is determined.

**The principle involved in gravimetric analysis by precipitation method are:**

- 1) A weighed sample is dissolved
- 2) Excess of precipitating agent is added in the solution containing sample.

- 3) The resulting precipitates are filtered and dried. They are weighed.
- 4) From the mass and known composition of the precipitates the amount of original ion can be determined.



$$\% \text{ of analyte} = \frac{\text{Wt of ppts} \times \text{Gravimetric factor}}{\text{Wt of sample}} \times 100$$

#### Gravimetric factor:

Atomic mass of  $\text{BaSO}_4 = 233.42 \text{ g/mol}$

Atomic mass of  $\text{Ba}^{+2}$  ion =  $137.36 \text{ g/mol}$

$$\text{Gravimetric factor} = \frac{137.36}{233.42} = 0.5885$$

- For the successful determination the following criteria must be met. The desired substance must be completely precipitated and solubility of precipitates must be low.
- The low solubility of precipitates is achieved by the excess of precipitating agent. For e.g.



Here, for decreasing solubility of  $\text{AgCl}$ ,  $\text{Ag}^+$  ions will be in excess.

- By decreasing the temperature of the solution, solubility of precipitates will be decreased (by using ice bath).
- The weighed form of the product should be of known composition.
- The product should be pure and easily filtered.
- It is usually difficult to obtain a product which is pure. But careful precipitation and sufficient washing helps reducing the levels of impurity.

#### Advantages of Gravimetric analysis:

- Analysis can be done accurately and precisely by using sensitive and modern analytical balance.

- Filtrate can be analysed for composition of the precipitation reaction and errors can be reduced to a great extent.
- Direct measurement (weighing) of the compound is done in gravimetric analysis, so calibration is not required (except for weighing balance) and it is an absolute method.
- It is cheap, easily available apparatus are used in this method and easily carried out.

#### Disadvantages of Gravimetric analysis:

- Gravimetric analysis usually provides for the analysis of a single element, or a limited group of elements at a time.

Comparing modern dynamic flash combustion with gas chromatography, it is the faster and allows for simultaneous determination of multiple elements. While, traditional determination allowed only for the determination of C & H.

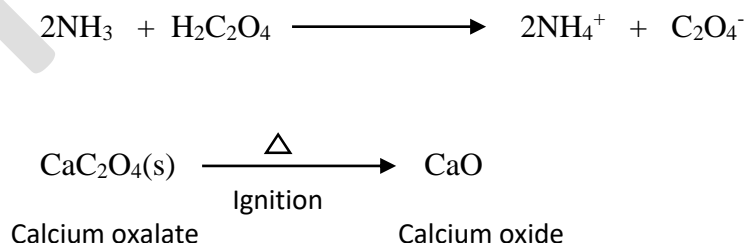
- This method is difficult (convoluted) and slight mis-step in a procedure can often mean disaster for the analysis (colloid formation in precipitation gravimetry). Compare this with hardy methods such as spectrophotometry and one will find that analysis by these methods is much more efficient.

### GRAVIMETRY BY PRECIPITATION

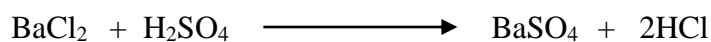
In precipitation gravimetry, analytes are converted into sparingly soluble precipitates, they are filtered and washed free of impurities. Then converted in to a product of known composition (by heat treatment). Finally product is weighed.

e.g. Determination of calcium from natural water. Excess amount of oxalic acid is added to sample solution. Calcium oxalate crystals are formed. Excess oxalic acid is neutralized by addition of  $\text{NH}_3$  solution.

Calcium oxalate crystals are collected, washed, dried and ignited. After ignition calcium oxalate is converted into calcium oxide, which is known as weighed form.



Sometimes, precipitated and weighed forms are same. Precipitates of  $\text{BaSO}_4$  are formed from  $\text{BaCl}_2$ .  $\text{BaSO}_4$  upon ignition remain such as  $\text{BaSO}_4$ .



### Conditions of precipitation

- Precipitates are of different types.
  1. Crystalline- They are well defined.
  2. Curdy- Aggregates of small porous particles.
  3. Gelatinous- Difficult to work with- Gelly like
- Filtration of precipitates can be easier, rapid and complete.

### Rules for precipitation:

- Precipitating agent should be mixed slowly and with constant agitation, which keep degree of supersaturation small and thus it will help in formation of crystalline precipitates.
- Precipitation must carried out in dilute solution.
- Precipitation should be carried out in hot solution, because precipitates are stable at higher temperature.
- Crystalline precipitates should be digested for longer time.
- Precipitates should be preferably washed with dilute solution of appropriate electrolyte. Precipitates should not be washed with water as it leads to peptization (Coagulated colloid reverts to its original dispersed state).
- Precipitates those are contaminated by co-precipitation should be dissolved in suitable solvent and reprecipitated.

### Properties of Precipitates:

- In gravimetric analysis precipitating reagent should be react specifically otherwise selectively.

#### Precipitates should fulfill following requirements.

- Precipitates should be insoluble. No significant loss occurs during filtration and washing.
- Physical nature of precipitates is such that they can be easily separated by filtration and be washed free of soluble impurities. In other words, particle should be of such size that they cannot pass through the filtering media and particle size is unaffected by washing.
- Precipitates should be stable to atmospheric conditions.
- Precipitate must be convertible to a pure compound of definite composition, which is possible by ignition or by simple chemical operation (evaporation).

Colloidal suspensions are not suitable in gravimetry. Stability of such suspensions is high. They cannot readily be filtered.

Stability can be decreased by stirring, heating and by adding an electrolyte, which causes individual colloidal particles to bind together to give colloidal mass.

The process of conversion of colloidal suspension into filterable mass is called coagulation or agglomeration or coalescence.

Colloidal particle size is  $0.1 \mu$  to  $1.0 \mu$ . Quantitative filter papers will retain particles up to  $10 \mu$ .

Colloidal suspensions are stable. All colloidal particles are either positively or negatively charged.

### **PURITY OF PRECIPITATES**

- When precipitates separate out from the solution, they are not always perfectly pure. Precipitates are contaminated with varying amounts of impurities even after extensive washing.
- Those amounts of impurities depend upon (1) Nature of precipitates  
(2) Condition of precipitation

#### **Factors affecting purity of precipitation:**

- 1) Co-precipitation
- 2) Post-precipitation

#### **Co-precipitation:**

- Some impurities get precipitated along with the substance to be analysed, although its solubility product is not exceeded. It would not have been precipitated if the analyte had been absent. This phenomenon is called as co-precipitation.
- In other words, Co-precipitation is defined as precipitation of any extraneous matter, which is not generally precipitated under general conditions by the precipitant used.
- It is often undesirable. Impurity of  $\text{KMgPO}_4$  is precipitated with analyte  $\text{NH}_4\text{MgPO}_4$ .
- For e.g. to a mixture of  $\text{BaCl}_2$ ,  $\text{KMnO}_4$  solution is added. Excess of  $\text{H}_2\text{SO}_4$  is added slowly. Here,  $\text{KMnO}_4$  is reduced. After the reaction, the solution becomes colourless and a precipitate appears to be violet, which indicates that some  $\text{KMnO}_4$  is co-precipitated with  $\text{BaSO}_4$  precipitates.
- **There are four types of co-precipitation**
  - 1) Surface adsorption
  - 2) Occlusion

- 3) Mixed crystal formation
- 4) Mechanical entrapment

1) **Surface adsorption:**

- Adsorption is a common source of precipitation. Significant contamination of precipitates occurs with large specific surface area. i.e. coagulated colloids.
- Adsorption does occur in crystalline solids. Its effect on purity is usually undetectable, because of its small specific surface area of these solids.
- Ions may adsorb on the surface of ionic substance (precipitates). These adsorbed ions form least soluble salt than original precipitates (of analyte). e.g. Calcium ion adsorbed more preferentially over Mg ions. Calcium sulphate is less soluble than  $MgSO_4$ .
- Adsorption is a reversible process. It is accompanied by the opposite process of desorption. i.e. passage of adsorbed ion from the surface of precipitate into the solution.
- Those two process adsorption and desorption occurs simultaneously. Equilibrium is maintained.

**Factors affecting surface adsorption:**

- **Effect of concentration:**  
Adsorption is directly related to concentration of those substances in solution. i.e. Adsorption increases with their concentration in solution.
- **Effect of temperature**  
Adsorption is an exothermic process. It is favoured by decrease in temperature. Rise in temperature leads to desorption and amount of substance adsorbed decreases.
- **Effect of nature of adsorbed ion**  
Adsorbents with ionic crystal lattices preferably adsorb ions which form sparingly soluble or weakly dissociated compound with oppositely charged ion in the lattice. i.e. particular ions common with precipitates.  
  
e.g. AgI precipitates adsorbs  $Ag^+$  and  $I^-$  ions.
- **Effect of precipitation conditions:**  
Adsorption is much affected by experimental conditions such as concentration of reacting solutions, temperature etc.
- **Effect of adsorbent area:**  
For crystalline precipitates, adsorption is insignificant. The ions or substances adsorbed on the surface of adsorbent is proportional to total surface area of the adsorbent surface.

Gelatinous precipitates have large surface area and hence adsorption will be greater, while adsorption is minimum on small surface area of crystalline precipitates.

## 2) Occlusion:

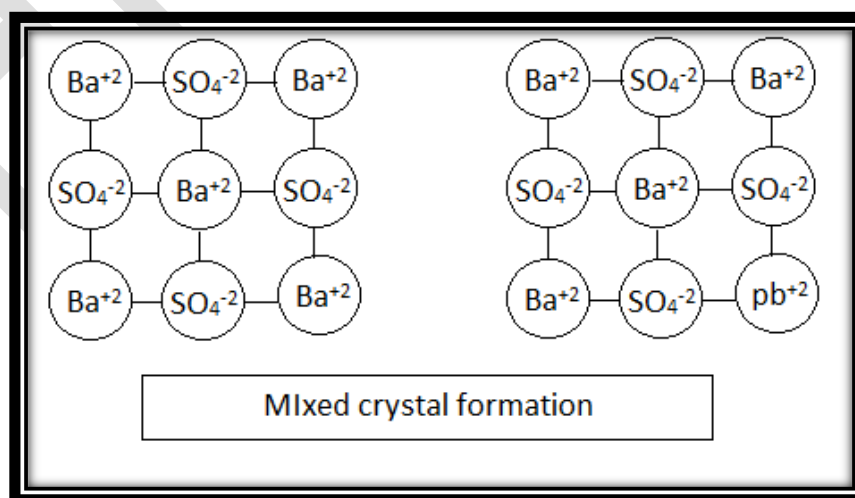
- When crystal is growing rapidly during precipitate formation, foreign ions in the counter ion layer may become trapped or occlude within growing crystals and form packet.
- If crystal growth rate is fastest, occlusion is more. As precipitation proceed, crystal growth decreases and rate of precipitation formation becomes low, thus occlusion is minimum.
- During coalescence of primary particles, impurities will eliminated if large crystal forms. Therefore, occlusion process takes place slowly.
- If coalescence is rapid, large crystal composed of loosely bound small crystals are produced. Some of impurities may be entrapped within the walls of large crystals they have no time to escape from the surface as the process of coalescence is rapid.
- Digestion may help to remove impurities. Digestion is to be done at elevated temperature. At elevated temperature, reprecipitation occurs which open the pockets and allows the impurities to escape into the solution.

## 3) Mixed crystal formation:

- In this, one of the ion in the crystal lattice of the solid is replaced by ion of another element.
- For this, 1) sizes of two ions are nearly same (difference should not be more than 5 %).

2) Charges are same.

For e.g.  $\text{BaSO}_4$  is precipitated by  $\text{BaCl}_2$ .  $\text{Ba}^{+2}$  ion is replaced by  $\text{Pb}^{+2}$ . Thus, mixed crystal formation occurs.



## 4) Mechanical entrapment: (solution entrapment between fast growing crystals)

- When crystals are close together during crystal growth, mechanical entrapment occurs.



- Several crystals grow together, it traps portion of solution in tiny packet.
- Rapid solution and reprecipitation at elevated temperature allows the impurities to escape into solution. This process gives better precipitates.

#### **Methods to minimize co-precipitation:**

To minimize surface adsorption, precipitate should be crystalline (larger particles). Due to larger particles crystalline precipitates have lesser surface area, thus absorption is also less. It gives more pure solution.

Colloidal particles which are small, they have larger surface area which promotes surface adsorption, therefore crystalline particles are preferred.

#### **Following are the methods to minimize co-precipitation**

- a) **Add precipitating agent** slowly with constant but slow agitation of solution. This will keep degree of super saturation small and help in formation of crystalline product.

The degree of super saturation is inversely proportional to particle size. As smaller degree of super saturation, larger particles are formed.

- b) **Digestion of precipitate**

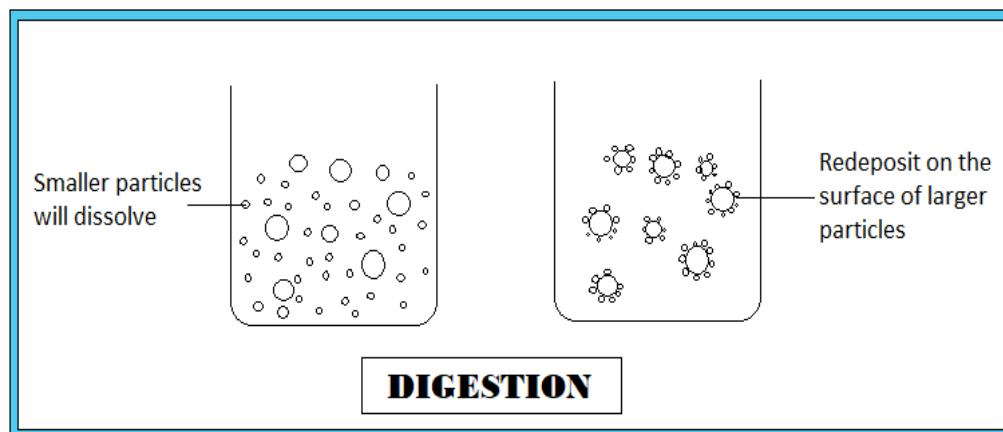
Generally, the primary precipitate obtained from a hot dilute solution is in the form of the nearly perfect crystal structure. However, those obtained from concentrated solutions are generally very small crystals of imperfect structure. There is considerable variation in particle size for any given primary precipitate.

When a precipitates is allowed to stand in presence of mother liquor (solution from which it is precipitated.), the large crystals grow at the expense of small ones. This phenomenon is called as **digestion** or **Ostwald ripening**.

In this small particles tends to dissolve because they have high solubility than larger one, smaller particles then re-precipitate on the surface of larger crystals. In addition, Individual particles will agglomerate to share a common counter ion layer and finally cement together to form large crystals.

Digestion is usually done at elevated temperature to speed up the process. In some cases, it is possible at room temperature also. This process helps to produce larger crystals that are more easily filtered from the solution.

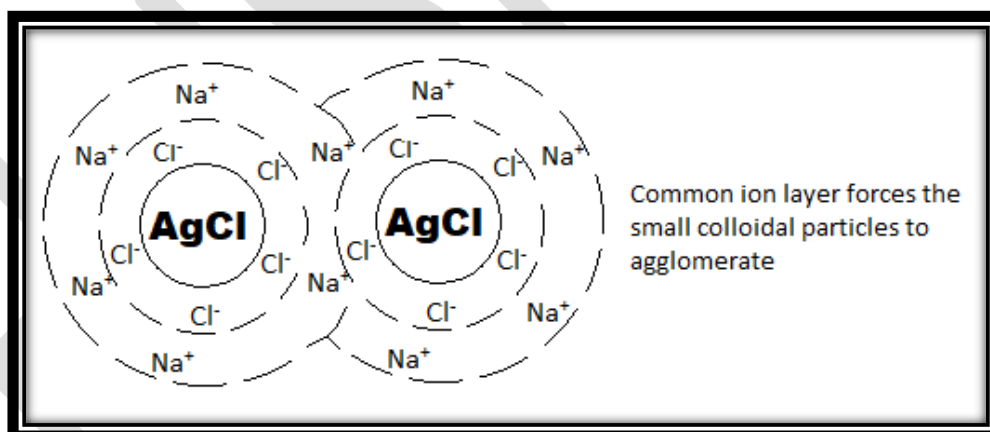
Small particles which have greater solubility and surface area will after precipitation tend to pass into the solution (small particles dissolve). These small particles ultimately re-deposit on larger particles, resulting into elimination of co-precipitation by surface adsorption.



On digestion, precipitates tend to become more regular, dense, crystalline particles and hence result in a decrease in surface area. Thus, digestion results in a reduction of surface adsorption (co-precipitation). Adsorption is a major problem in gravimetry.

Digestion reduces the extent of co-precipitation and increases the size of crystals of precipitates, which facilitates the filtration.

Digestion forces the small colloidal particles to agglomerate, which decreases their surface area and thus adsorption. Colloidal precipitates tend to adsorb their own ions (present in excess) which form a primary ion layer followed by the formation of a secondary (counter-ion) layer.



#### 🚩 Post precipitation:

- Sometimes when the precipitates are allowed to stand in the presence of mother liquor, the second substance which forms the precipitate with the precipitating reagent is called post-precipitation. It generally occurs with sparingly soluble substances which form a supersaturated solution.
- They usually have an ion in common with the primary particles of precipitates. e.g. calcium oxalate is precipitated in the presence of magnesium ions, magnesium oxalate does not precipitate immediately, because it tends to form a supersaturated solution.

- If these precipitates are allowed to stand for longer time before filtration, magnesium oxalates separates out on precipitate of calcium oxalate.
- Longer the precipitates of analytes remain in the solution, greater will be the error due to post precipitation.
- Post precipitation is minimized by (1) filtration of precipitation of analyte as soon as possible  
(2) By agitation of solution

### Difference between co-precipitation and post precipitation

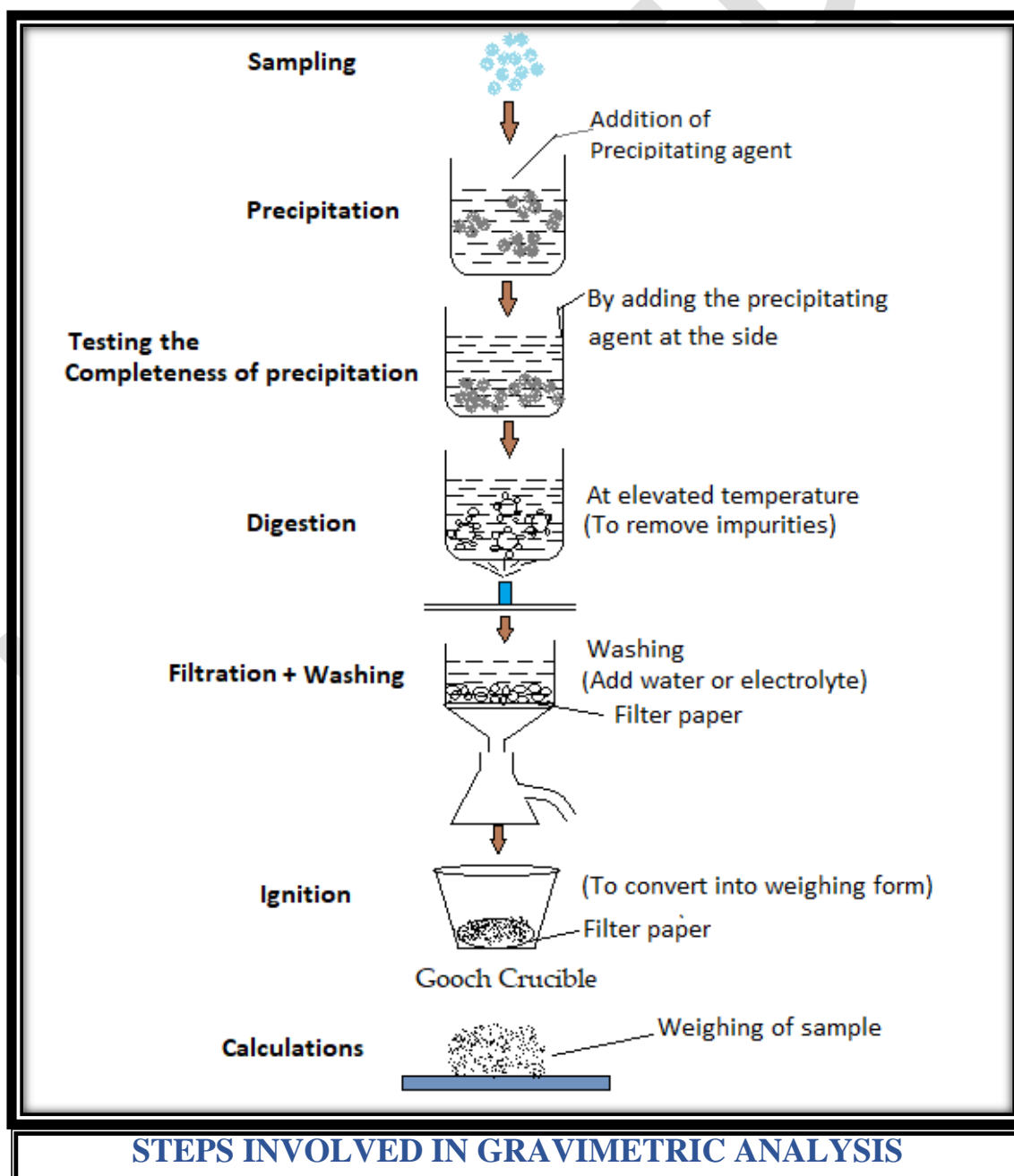
Co-precipitation	Post precipitation
It occurs because impurities are present in solution in soluble form. It occurs easier during isolation of precipitation of analyte.	It occurs in second stage. i.e. when precipitation is left for longer time in mother liquor.
Decreases contamination by impurities by co-precipitation with agitation by mechanical or thermal means.	The extent of contamination increases with faster agitation by mechanical or thermal means in case of precipitation.
The extent of contamination decreases with the time of contact with mother liquor in case of co-precipitation.	In post precipitation, extent of contamination increases with the time of contact with mother liquor.
If precipitation digest for longer time at elevated temperature on steam, the co-precipitation decreases.	If precipitation digest for longer time at elevated temperature, post precipitation increases.
The extent of contamination by co-precipitation is low.	The extent of contamination by post precipitation is much higher than co-precipitation.
<b>Example:</b> Contamination of $\text{BaSO}_4$ by $\text{KMnO}_4$ impurities which is present in the solution in the soluble form.	<b>Example:</b> Contamination of calcium oxalate precipitates in presence of $\text{Mg}^{+2}$ because it reacts slowly with oxalic acid. After that mg-oxalate is precipitated.

### STEPS INVOLVED IN GRAVIMETRIC ANALYSIS

The operation of gravimetric analysis consist of number of important techniques in order to get accurate and quantitative estimation. It include:

- 1) Sampling

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



**1) Sampling:**

- The sample for analysis is very small. It is necessary to take representative sample correctly to ensure that results are accurate.
- The sample should be homogenous and in form of powder.
- Ideal sample would be identical in all its properties with the bulk of the material from which it is taken.
- Cost of test, value of product, end use of products, accuracy of test method and nature of materials used are considered.

**2) Dissolution:**

- To dissolve the sample take a clean beaker and transfer the weighed sample completely to the beaker. Add sufficient water to the sample to get clear solution.
- Solution condition must be adjusted to have low solubility of the precipitate and to obtain it in the suitable form for filtration.
- Proper adjustment prior to the precipitation may mask the interference. The other factors to be considered are temperature, pH, and concentration of other constituent and volume of the solution.
- If the substance is soluble only in acids or alkalis, this reaction evolve gases. Thus, such reaction allows to take place to cease the evolution of gases. If heating is necessary for dissolution, then heat the solution on water bath.

**3) Precipitation:**

- Proper choice of precipitant is based on the specificity of reagent. If specific reagent is not possible, it should be selective.
- For ideal precipitating agent, It should react with analyte to form precipitates which can be—
  - easily filtered,
  - washed free of contaminating impurities
  - should have low solubility
  - Should be stable and should have constant known composition after drying.
- Precipitation are generally carried out in resistant glass beaker with slow addition of dilute solution of precipitates with sufficient stirring.
- This solution should be carried out in hot solution. (Precipitates are stable at hot temperature.)
- For proper precipitation, a moderate excess of precipitant is required. A very large excess of precipitant may lead to increase in solubility.

- After precipitates have settled down, add a few drops of precipitant to the solution to check completeness of precipitation.
- Avoid the boiling of the solution to avoid the losses due to spattering.
- If the ion being precipitated can be precipitated by various precipitants, the precipitant which forms a precipitate having the least solubility should be chosen.

#### 4) Testing the completeness of precipitation

- In gravimetry analysis, the completeness of precipitation is checked by adding a few drops of precipitant by side of the beaker after all precipitates have been settled down.
- Effect is to be checked in the solution when one drop of precipitating agent is added. If no turbidity is seen at the edge, it shows that precipitation is complete.
- It is always advisable to check the completeness of precipitation before the filtration.

#### 5) Digestion

- Digestion is generally carried out at higher temperature to speed up the process, but in some cases, it is done at room temperature.
- It improves the purity and rate of filtration of precipitate. The surface adsorption is minimized by digestion.

#### 6) Filtration

- In this operation, precipitate is separated from mother liquor. Various types of filter media are used for this purpose.
- The choice of filter media is based on the nature of precipitate, cost of media and heating temperature required for drying.
- The filtering medias used in this are:
  1. Filter paper
  2. Filter Pulp
  3. Filter mats
  4. Permanent porous filter discs

- I. Filter paper:** Quantitative filter paper used in gravimetric analysis should have low ash value. This ash value can be estimated by ignition of several papers in the crucible. Lower value of ash content are usually achieved by washing with hydrochloric acid, hydrofluoric acid during its manufacture.

Circular filter papers with different degrees of porosities like 7.0, 9.0, 11.0, & 12.5 cm diameters are available

For gelatinous and coarse particles	Medium size particles	Very fine particles
<u>Fast speed</u>	<u>Medium speed</u>	<u>Slow speed</u>
No. 31 (4.6 $\mu$ )	No. 30 (2.2 $\mu$ )	No. 32
No. 41 (4.1 $\mu$ )	No. 40 (2.4 $\mu$ )	No. 42
No. 54	No. 52	No. 50

Filter paper treated with Nitric acid have greater mechanical strength and low ash value. No. 42 is used for fine particles. No. 41 is used for gelatinous precipitates.

## II. Filter pulp: (for gelatinous precipitates and more fine particles)

For gelatinous precipitates filter papers are not generally used, because pores of filter paper are clogged by gelatinous precipitates. Thus, filter pulp is used instead of filter paper for filtration.

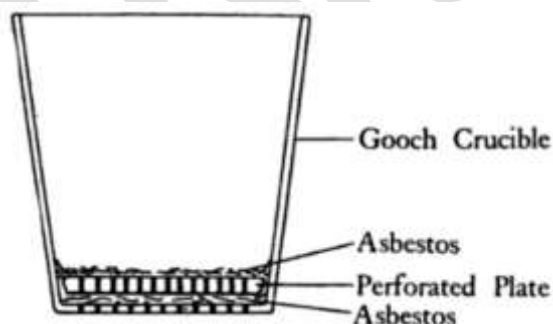
Filter pulp can be prepared by macerating the filter paper torn into pieces with hot distilled water. Watt man filter clippings are also used for this purpose.

The precipitate along with filter pulp is transferred to crucible, when it is still wet and drying is completed in crucible.

For every fine particles which are difficult to coagulate can also be filtered by using filter pulp.

## III. Filter Mats:

- 1) **Gooch Crucibles:** Bottom of the crucible was perforated with numerous small holes.



## 7) Washing of precipitates:

- After filtering the precipitate; the impurities on the surface of precipitate can be removed by washing of precipitate. Mother liquor which is entrapped with precipitate is also removed by washing.

- Many precipitates are not washed with water, but it can be washed with other solution such as electrolytes. Ideal washing solution should have no solvent action on precipitates but should dissolve foreign matter easily.
- The washing solutions are of following types-

**a) Washing with the solution of precipitant:**

In this easily the precipitate is washed with dilute solution of precipitant. e.g. Lead sulphate precipitate washed by dilute sulphuric acid.

**b) Washing with solution of electrolyte:**

When pure water is used for washing the precipitate, many precipitates may be peptized (revert back to disperse) and converted to colloidal state. Therefore washing is done with electrolyte solution.

e.g. Washing of silver chloride precipitate with dilute solution of nitric acid.

**c) Washing with substance which suppress hydrolysis of precipitates:**

Sometimes precipitates undergo the hydrolysis leading to increase in solubility. Thus, hydrolysis is prevented by washing with the solution which suppress hydrolysis. e.g. washing of  $\text{MgNH}_4\text{PO}_4$  precipitates with dilute solution of  $\text{NH}_3$ .

**d) Washing with water:**

If the precipitate is very stable and therefore is no effect of solubility or no effect leading to peptization or no effect leading to hydrolysis of precipitates, in these situations precipitates can be washed with distilled water. e.g. Washing the precipitate of  $\text{BaSO}_4$  with water.

**8) Drying and Ignition of precipitates:**

- The main object of drying is to convert the precipitate to a weighed form and having constant and definite composition.
- It is heated to remove water and adsorbed electrolyte from washing solution.
- Drying and ignition process will depend upon the nature of precipitate and the filtering media used for the purpose.
- Drying is the term used when temperature is below  $250\text{ }^\circ\text{C}$  and ignition above  $250\text{ }^\circ\text{C}$  and below  $1200\text{ }^\circ\text{C}$ . Drying is possible for precipitates filtered on filter paper, Gooch crucibles and sintered glass crucibles.
- Ignition is done by heating with appropriate burner or crucible which can be placed in electrically heated muffle furnace. Heating to constant weight is an important aspect. It will be significant only when period of heating, cooling of covered crucible and weighing are repeated.



- Precipitates can be ignited in contact with filter paper or without filter paper. Separate carefully filter paper from precipitates.

### 9) Weighing and Calculations:

$$\% \text{ of analyte} = \frac{\text{Wt of ppts} \times \text{Gravimetric factor}}{\text{Wt of sample}} \times 100$$

### ESTIMATION OF BARIUM SULPHATE

☆ In gravimetric analysis, the amount of sulphate is estimated quantitatively as barium sulphate (BaSO<sub>4</sub>). In this assay, dilute solution of barium chloride is gradually added to a hot acidic solution of sulphate. This solution is acidified with the hot hydrochloric acid solution.



☆ The barium sulphate precipitate is filtered, washed with water, ignited at red heat, and weighed.

#### Procedure:

- 1) A porcelain crucible is heated to redness and placed in a desiccator to cool.
- 2) Accurately weighed 0.2 gm of barium chloride is dissolved in 100 ml of water.
- 3) The solution is boiled and a slight excess of hot 0.5M sulphuric acid solution is added slowly in it with continuous stirring. On a steam bath, the solution is digested until the precipitate has settled.
- 4) Few drops of 0.5 M sulphuric acid are added to the supernatant that confirms the completion of precipitate.
- 5) For filtration a Whatman (No. 40) filter paper is positioned on a funnel.
- 6) The precipitate is transferred to the filter paper using a jet of warm water from the wash bottle.
- 7) The precipitate is washed a number of times using small portions of hot water. Washing is continued till about 5 ml of the filtrate shows no turbidity with one or two drops of dilute AgNO<sub>3</sub> solution.
- 8) The cooled crucible is weighed and filter paper is transferred to the crucible with the precipitate.
- 9) The paper is kept for drying with the loosely covered crucible over a low flame. Heat is increased slowly till the paper chars and the volatile matter is expelled. The whole process should be performed carefully to avoid the bursting of paper into flame.

- 10) After the completion of charring, temperature is increased till the crucible becomes dull red in colour, and burn off the carbon with free access of air.
- 11) The crucible is kept for 10 minutes at red heat, after the precipitate turns white.
- 12) The crucible is placed in desiccator for cooling and weighing is done.
- 13) The process of heating and cooling is repeated continuously till a constant weight is obtained.
- 14) The percentage of barium is calculated in the sample.

$$\% \text{ of analyte} = \frac{\text{Wt of ppts} \times \text{Gravimetric factor}}{\text{Wt of sample}} \times 100$$

**Gravimetric factor:**

Atomic mass of BaSO<sub>4</sub> = 233.42 g/mol

Atomic mass of Ba<sup>+2</sup> ion = 137.36 g/mol

Gravimetric factor = ( 137.36 )/233.42 = 0.5885