

Unit - II

Gravimetric Analysis**Principle :**

Gravimetric analysis is an analytical technique by which one estimates the amount of a substance present in a given sample by determining the weight of precipitate obtained from the sample.

Example :

To determine the amount of chloride present in a given solution it is treated with enough AgNO_3 solution to precipitate the chloride completely. The precipitate is filtered off, dried and weighed. From the weight of AgCl precipitate, amount of chloride present in the whole of the given solution is calculated.

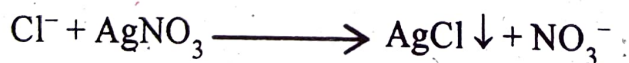
Gravimetric factor :*Definition :*

$$\text{Gravimetric factor} = \frac{a}{b} \times \frac{\text{Formula weight of substance to be estimated}}{\text{Formula weight of substance to be weighed}}$$

Where a and b are integers that make the number of formula weights in the numerator and denominator chemically equivalent.

Explanation with examples :

i. Let us take the example of estimation of chloride ion. It is converted into AgCl with the help of AgNO_3 . The relevant balanced equation is



Here as per equation one gram ion of Cl^- is converted into one mole of AgCl . So here $a = 1$ and $b = 1$.

Formula weight of substances to be estimated = formula weight of $\text{Cl}^- = 35.45$.

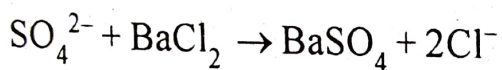
Formula weight of the substances weighed = Formula weight of AgCl
 = $107.87 + 35.45 = 143.32$.

For the estimation Cl^- as the AgCl the gravimetric factor is

$$= \frac{1 \times 35.45}{1 \times 143.32} = 0.2473$$

When this gravimetric factor is multiplied by the weight of AgCl precipitate obtained during the gravimetric estimation, the amount of the Cl^- is obtained.

ii. Let us take the example of estimation of sulphate ion. It is converted into BaSO_4 with the help of BaCl_2 . The relevant balanced equation is



Here also, as per equation one gram ion of SO_4^{2-} is converted into one mole of BaSO_4 . So here also $a = 1$ and $b = 1$.

Formula weight of the substance to be estimated = formula weight of $\text{SO}_4^{2-} = 96.06$.

Formula weight of the substance weighed = Formula weight of $\text{BaSO}_4 = 137.34 + 32.06 + (16 \times 4) = 233.4$.

For the estimation SO_4^{2-} as the BaSO_4 the gravimetric factor is

$$\frac{1 \times 96.06}{1 \times 233.34} = 0.4117$$

When this gravimetric factor is multiplied by the weight of BaSO_4 precipitate obtained during the gravimetric estimation, the amount of the SO_4^{2-} is obtained.

Characteristic of precipitating agent :

A reagent added to a solution of a substance to be estimated. So, as to precipitate that substance is called a precipitating agent. E.g., To estimate barium in a solution of chloride we use potassium chromate solution. Thus, potassium chromate solution is the precipitating agent or the precipitant.

An ideal precipitating agent should react specifically with the ion or substance to be estimated gravimetrically and give a precipitate.

- i. The precipitate has sufficiently low solubility so that losses, due to solubility of the solid is negligible.
- ii. Which is readily filtered and washed free of contaminants.
- iii. Which is unreactive and of known composition after drying or if necessary ignition.

Choice of precipitants :

Precipitating agents may be inorganic or organic. Some inorganic precipitating agents used are

- i. Barium chloride to estimate sulphate ion
- ii. H_2SO_4 or K_2CrO_4 to estimate Ba^{2-} ions.
- iii. HCl to estimate Ag^+ ions
- iv. AgNO_3 to estimate Cl^- ions etc.

Some organic reagents used are

- i. DMG (dimethyl glyoxime) to estimate Ni^{2+} ions.
- ii. Oxine (or 8 - hydroxy quinoline) to estimate aluminium etc.

The choice of precipitants for estimating metal ions is limited ; as only one or two precipitants are there which satisfy all the requirements of a good precipitant. For example, for estimating barium we have H_2SO_4 or K_2CrO_4 only. However we can choose between an organic precipitant and an inorganic precipitant. For example to estimate aluminium we can use ammonium hydroxide or oxine as precipitant. While using inorganic precipitants we may come across co - precipitation or post precipitation.

(Discussed later) Use of organic precipitants has both advantages and disadvantages.

Advantages of organic precipitants :

1. They give precipitates which are insoluble in water. So quantitative precipitation is possible.
2. The formula weight of the precipitative is large with respect to the formula weight of the metal ion to be estimated. This decreases the error component in weighing.
3. They give precipitates with large particle size. This makes filtration and washing efficient.
4. Drying of precipitates is easy as organic compounds have least tendency to retain water in them.

Disadvantages :

1. Drying makes the composition of the precipitate uncertain as organic substances are decomposed or volatilise on heating.
2. Preparation of the solution of the precipitating agents is difficult as the solubilities of these reagents in water is less. E.g., The solution of DMG has to be prepared in a cool as it is insoluble in water.

Specific precipitants :

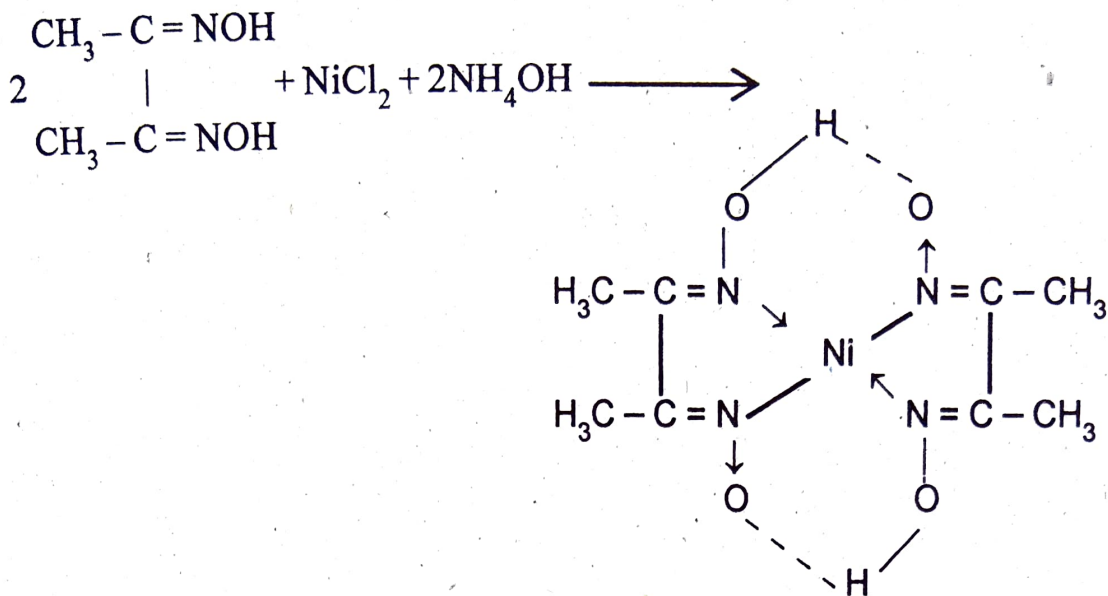
Specific precipitants precipitate quantitatively a particular ion or a substance from a solution.

There are no specific precipitants in reality. However some precipitants may be mentioned which are nearly specific.

Examples :

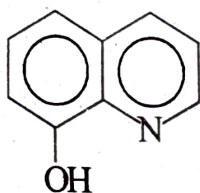
DMG (Dimethyl Glyoxmine) :

It is specific precipitant for estimating nickel (in alkaline medium) and palladium (in acid solution). Its reaction with Ni^{2+} ions is given below ;

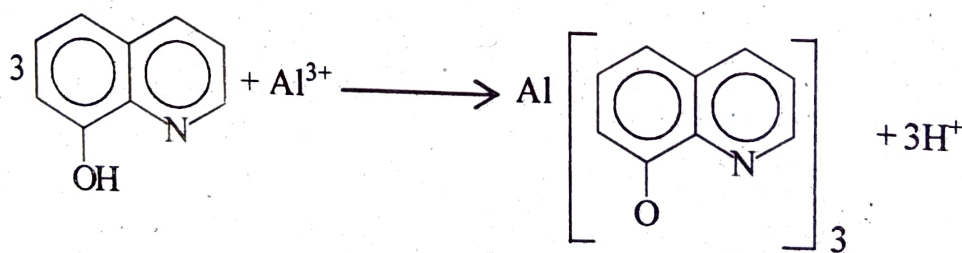


Nickeldimethyl glyoxime
Bright red precipitate

Oxine (8 - Hydroxy quinoline)



About 24 cations give precipitates with oxine. It is used to estimate aluminium from a solution with pH around 3, its reaction with Al^{3+} ions is given below ;

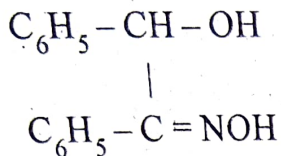


Some other metals that are precipitated by oxine are Mg, Cu, Zn etc.

The solubilities of metals oxinates vary from cation to cation and are pH dependent. So through pH control a considerable degree of selectivity can be achieved.

Cupron (Benzoin - α - Oxime) :

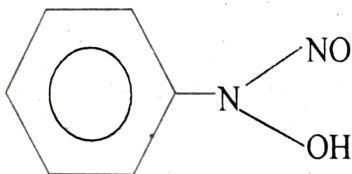
It is used to estimate copper in dilute ammoniacal solution in presence of tartrate.



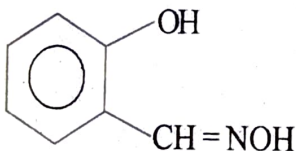
A green precipitate is got, it is dried at 110°C and weighted.

Cupferron :

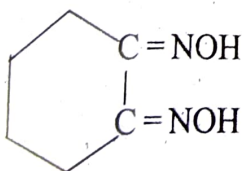
It is the ammonium salt of N - nitroso - N - Phenyl hydroxyl amine. It is used to estimate Cu(II) and Fe(III) in cold aqueous acid medium. Its structure is

**Salicyladehyde Oxime :**

It is also used to estimate Cu(II) in presence of acetic acid at pH 2.6.

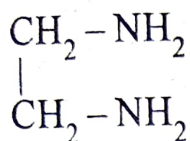
**Nioxime :**

It is cyclohexane 1, 2 - dione dioxime. It is used to estimate palladium. It is more than DMG.



Ethylene Diamine :

It is 1, 2 diamino ethane. It is used to estimate Cu(III), Hg(II) and Cd(II) ions.



Selective precipitants :

Selective precipitants precipitate a small group of ions from solutions containing several ions. Most of the gravimetric precipitants are only selective.

Example ;

OH^- precipitates a few metal ions as their hydroxides from a solution containing several metal ions. The metal ions which are not precipitated also from hydroxides but they are soluble and so are not precipitated S^{2-} , CO_3^{2-} are some other examples of selective precipitants.

When controlled conditions are adopted, selective precipitants may be increased.

Condition of precipitation

1. The ion or substance to be estimated gravimetrically should be precipitated quantitatively so that the filtrate does not contain that substance which is precipitated i.e., solubility must be very low.
2. The precipitate must be in the form of large particles so that it is readily filtered.
3. The precipitate must be pure. It should not be contaminated with their substances. Even if it contains some impurities they must be removable by washing and drying.
4. The precipitate must have a constant composition, i.e., it should not change its composition during filtration, drying or if necessary ignition.

Techniques of precipitation :

To fulfil the conditions of precipitation the following techniques are adopted.

1. The solution of analysis (substance to be estimated) is diluted. This will enable us to precipitate the analyte quantitatively.
2. The precipitant added must be chosen in such a way that it forms.
3. Precipitation must be done in hot condition. In hot solution the solubility increases reducing super saturation. Precipitation is hastened. Colloid formation is avoided. We get well formed precipitate.
4. The precipitant should be added slowly and in small lots with constant stirring. This will lead to quantitative precipitation of the analyte.
5. Precipitation is done from homogeneous solutions so that supersaturation is minimized and help the growth of the size of the precipitate.
6. Slight excess of the precipitant should be added to ensure quantitative precipitation.
7. The precipitate is to be digested for sufficiently long time to give precipitate particles of considerably big size so that it is not lost during filtration.
8. The precipitate should be washed with suitable wash liquid (containing suitable electrolyte) to avoid peptization (dissolution of some precipitate). If the precipitate is suspected to contain impurities because of co - precipitation etc., It should be dissolved in a suitable solvent and reprecipitated again.

Types of precipitates :

Precipitates are of two types : i. Colloidal, ii. Crystalline.

i. Colloidal precipitates :

Individual colloidal particles are so small that they are not retained on ordinary filtering media. Further more, Brownian motion prevents their setting from the solution under the influence of gravity. But the individual

particles of most colloids can be coagulated to give a filterable, non crystalline mass that rapidly settles down from solution.

Heating, stirring and adding an electrolyte to the medium hastened the coagulation process.

ii. *Crystalline precipitates :*

Crystalline precipitates are more easily manipulated than coagulated colloids. The size of individual crystalline particles can be varied to a degree.

Formation of precipitate Nucleation and crystal growth

Theory / Mechanism :

There are two competing precipitation mechanisms

- i. Nucleation
- ii. Crystal growth.

The size of the precipitate depends on the fact whether i. or ii. dominates.

i. Nucleation

Nucleation is process in which four or five ions or molecules unite to form a single particles called a nucleus. Further precipitation occurs either by generation of additional nuclei or by the deposition of more and more of ions or molecules on the nuclei that have already been produced. If nucleation predominates, a precipitate containing a large number of small particles result. If particle growth predominates, the precipitate containing small number of large particles result.

ii. Crystal growth

Factors affecting the particle size of a precipitate.

Particle size depends on

- i. Chemical composition of a precipitate
- ii. The existing conditions during its formation.
- iii. Solubility of the precipitate in the medium in which it is being formed

- iv. Reactants concentration
- v. The rate at which the reactants are mixed
- vi. Temperature at which the precipitation is conducted are some of the factors which influence the particle size of the precipitate. The effect of these factors can be accounted for qualitatively, by assuming that the particles size is related to the relative supersaturation of the system.

$$\text{Relative supersaturation} = \frac{Q - S}{S}$$

Where Q = concentration of the solute at a given instant, S = Its equilibrium solubility. When $(Q - S)/S$ is large, the precipitate tends to be colloidal. When this parameter is small we get coarse, crystalline particles.

Conditions to get precipitate in the form of large particles :

- i. Reprecipitation is to be conducted at elevated temperature, this increases S
- ii. Precipitation is done from dilute solutions. This decrease Q
- iii. The precipitating agent is to be added slowly. This also decrease Q .
- iv. While adding the precipitant, the solution is to be stirred well. This also decreases Q .

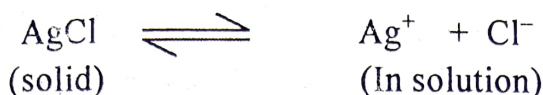
Solubility Product :

Definition :

The solubility product of a sparingly soluble salt is the product of the concentrations of ions in the saturated solution. It is a constant at a given temperature. It is denoted as K_{SP} .

Explanation :

When a slightly soluble ionic solid such as silver chloride is placed in water. Ag^+ ions and Cl^- ions from the solid phase pass into solution till the solution becomes saturated. Now there exists an equilibrium between the ions present in the saturated solution and the solid phase. Thus



Applying the law of chemical equilibrium, the value of equilibrium constant.

$$K = \frac{a_{\text{Ag}^+} a_{\text{Cl}^-}}{a_{\text{AgCl}}}$$

Since the activity of a solid is taken as unity the above expression may be written as

$$K a_{\text{AgCl}} = a_{\text{Ag}^+} a_{\text{Cl}^-} \text{ (or)}$$

$$K_{\text{sp}} = a_{\text{Ag}^+} a_{\text{Cl}^-}$$

K_{SP} is called the solubility product. It is a constant at a given temperature.

Ionic Product :

Ionic product of a substance in solution is the product of the concentrations of its ions in that solution.

Explanation :

When an ionic solid is dissolved in water, it dissolves. It ionises in solution. Now, the product of the concentration of the ions present in the solution is the ionic product of that substance.

Explanation with example :

Let us take an ionic solid like NaCl. When it is dissolved in water it ionises as follows ;



Let the concentration of Na^+ be $[\text{Na}^+]$ and that of Cl^- be $[\text{Cl}^-]$. Now the ionic product of NaCl in that solution is given by

$$\text{Ionic product of NaCl} = [\text{Na}^+][\text{Cl}^-]$$

Solubility product principle :

Principle :

When the ionic product of a substance in its solution exceeds its solubility product it is precipitated.

Explanation :

We know that solubility product of a substance is actually the ionic product of that substances in its saturated solution. So when the value of the ionic product of a substance in its solution exceeds its solubility product, it is precipitated, because it contains more substance than necessary to make it a saturated solution.

Application / use in gravimetric analysis :

We use this solubility product principle in our gravimetric analysis.

A slight excess of the precipitating agent is added to the solution of the analysis so that the ionic product of the substance to be estimated in solution exceeds its solubility product and gets precipitated quantitatively.

Purity of Precipitates :

The purity of a precipitate is very important for accurate gravimetric estimation. The precipitate may be contaminated by one or more substances present in the solution during precipitation.

Methods by which contaminations occur :

1. There are negative and positive centers on the surface of the precipitate. Positive and negative ions from the mother liquid may get adsorbed on these centers respectively.

The nature of such adsorption is given by a law called Paneth - Fajans - Hahn law. The law is a precipitate preferentially adsorbs that ion which forms the least soluble compound with an ion present in its lattice. Eg., Calcium oxalate preferentially adsorbs magnesium ions than sodium ions since magnesium oxalate is less soluble than sodium oxalate.

2. As a crystal is formed during precipitation some impurities may be imbibed with in the growing lattice.

Co - precipitation :**Definition :**

It is a process by which normally soluble components of a solution are carried down during the formation of a precipitate.

Explanation with example :

In the determination of barium ions by precipitation as barium sulphate other barium salts such as barium nitrate and barium chloride occlude on barium sulphate precipitate. We know barium nitrate and barium chloride are soluble. Still during the precipitation of barium sulphate, these soluble compounds occlude on barium sulphate particles and are carried down along with barium sulphate precipitate. Such a process is known as co - precipitation.

(Contamination of precipitate by a second substance which is also precipitated along with the substance to be precipitated does not constitute co - precipitation)

Theory / Mechanism of co - precipitation :

Co - precipitation occurs either by surface adsorption, inclusion or occlusion. Gelatinous precipitates co - precipitate by adsorption while crystalline precipitates co - precipitate by inclusion or occlusion. Adsorption is a surface phenomenon. Inclusion involves the random distribution of the contaminant throughout the interior of the solid. Occlusion involves a non - homogenous distribution of the contaminant within the imperfections in the crystal lattice of the precipitate.

Role / Disadvantage / Effect of Co - precipitation :

Because of co - precipitation positive or negative error may result depending on whether the co - precipitated substance has more or less molecular weight than that of the precipitate. For example, during the precipitation barium sulphate, if barium nitrate is co - precipitated, a positive error is observed as the molecular weight of barium nitrate is more than that of barium sulphate. On the other hand, if the barium chloride is co - precipitated, a negative error arises as the molecular weight of barium chloride is less than that of barium sulphate.

Methods to decrease or minimise co - precipitation :

1. The solution, from which precipitation is carried out, should be diluted.
2. The precipitant is to be added in small quantities with constant stirring so that the precipitation takes place as slowly as possible.

3. Precipitation is carried out from hot solution only so that, the solubility of the impurity would be increased, formation of colloidal particles would be decreased and the attraction between the precipitate and impurity would be decreased.
4. The precipitate is digested well. These methods would decrease co-precipitation due to surface adsorption.
5. The precipitate is dried at high temperatures so that the occluded impurity could escape.
6. Solutions employed in precipitation are well diluted so that when the mother liquor adheres to the precipitate there is no much solute in it. This will decrease occlusion.
7. Crystal growth must be slow so that occlusion is less.

Post Precipitation :

Definition :

It is a process in which an impurity is precipitated from its supersaturated solution some time after the appearance of the substance to be estimated as precipitate.

Explanation with example :

When calcium is determined as its oxalate any magnesium present in the solution will be precipitated slowly on calcium oxalate. Similarly when Cu^{++} or Hg^{++} is determined as its sulphide, any Zn^{++} present in the solution will be precipitated slowly on CuS or HgS . Post precipitation becomes pronounced when the precipitate is permitted to be in contact with the mother liquor.

Theory of post - precipitation :

It occurs with sparingly soluble substances which form supersaturated solutions. These substances usually have an ion in common with the analyte. E.g., post precipitation of magnesium oxalate while estimating calcium by precipitating it as calcium oxalate.

Effect of post precipitation :

It will lead to positive error.

Remedy / Methods to decrease post - precipitation :

1. The precipitate is washed with suitable solution so as to remove the post precipitated impurity.
2. The precipitate should be filtered off as soon as the precipitation is over.

Differences :

<i>Co - precipitation</i>	<i>Post - precipitation</i>
1. It decreases with time when the precipitate is allowed to be in contact with its mother liquor.	it increases
2. It decreases when the solution is stirred or heated.	it increases
3. The amount of the substance co-precipitated is far less.	it is much more

Precipitation from homogenous solution

Definition :

This is a process in which the precipitant is slowly and chemically generated in the reacting solution itself.

Explanation / Illustration with example :

Ba^{2+} is precipitated as $BaSO_4$ by mixing Ba^{2+} solution and a solution containing a calculated excess of sulphamic acid. Sulphamic acid solution slowly generates SO_4^{2-} ions in solution by hydrolysis.



These SO_4^{2-} ions react with Ba^{2+} ion and form $BaSO_4$ which is precipitated. Dimethyl sulphate is also used to precipitate Ba^{2+} .

Uses / Advantages :

Precipitation from homogeneous media is done to avoid local reagent excesses, as the precipitating agent appears slowly and homogeneously, throughout the entire solution. The relative supersaturation is thus kept low. This method results in marked increase in particle size of the precipitate and its purity.

Digestion of precipitate :

It is the process of allowing the precipitate for several hours (some time even for a day) at room temperature or at a desired higher temperature in contact with the solution from which it was formed.

Effect of digestion :

1. Digestion promotes complete precipitation in a form which can readily be filtered.
2. During digestion small particles will tend to pass into solution and then redeposit on the larger particles. Thus it promotes the formation of a regular crystal shape with lesser surface area which in turn reduces co - precipitation by adsorption.
3. During digestion the impurities trapped inside the mass of the precipitate escape from their places. Thus the purity of the precipitate increases.

Washing of precipitate :

The digested precipitate contains some other substances as impurity on its surface. They are removed by washing the precipitate with wash liquids or solution in which the impurities are soluble. Washing removes the surface impurities only. Usually pure water is not used for washing as it would peptise the precipitate. i.e., some precipitate would go into the solution thus leading to the loss of some precipitate resulting in negative error.

A solution, containing an electrolyte having a common ion with the precipitate and which volatilizes when the precipitate is dried, is used for washing the precipitate. This reduces errors due to peptization. Some commonly employed wash solutions are dilute acids, ammonium salts solutions and ammonia solution.

Hot wash solutions are used if the precipitate is insoluble in hot solutions. This speeds up washing process as the impurities are more soluble in hot solutions there by decreasing the time taken for filtration.

Types of wash solutions :

The are three types ;

1. Solutions that prevent the precipitate from passing into the colloidal state and there by passing through the filter E.g., Ammonium salt solutions.
2. Solutions that reduce the solubility of the precipitate. E.g., Calcium oxalate precipitate is washed with ammonium oxalate solution. This is because calcium oxalate is less soluble in ammonium oxalate. Since most of the precipitates are insoluble and some impurities are soluble in ethanol or ether, they are sometimes used as wash liquids.
3. Solutions that prevent hydrolysis of the precipitate E.g., Aqueous ammonia is used to wash MgNH_4PO_4 because the latter hydrolyses to HPO_4^{2-} and OH^- ions.

Precautions to be followed while washing :

1. Least quantity of wash solution is to be employed to minimise loss of precipitate due to washing.
2. Many small portions of the wash solution are to be employed than a few large portions. Eg., Let us say that we want to use 100 ml of wash solutions. It is better to wash the precipitate four times with 25 ml of the wash solution each time, rather than two times with 50ml of the wash solution.
3. The filtrate is to be tested for the absence of expected impurity and the washing is stopped at an appropriate time. E.g., In the estimation of AgCl , the wash liquid is dilute HNO_3 . The washing is to be continued till the filtrate is free from Cl^- ions.

Requirements of a good wash solution :

- i. It should dissolve the impurity and should not dissolve the precipitate.
- ii. It should not disperse i.e., it should not peptise the precipitate.
- iii. It should not form any volatile insoluble product with the precipitate.
- iv. It should be easily volatilized at the drying temperature of the precipitate.

Ignition of the precipitate :

The precipitate is to be ignited / dried at a suitable temperature. It should be ignited / dried at a temperature which is sufficiently high to volatilize water, wash liquid and other volatile impurities, burn out the filter paper completely but must be well below the decomposition temperature of the precipitate. This is ascertained from a study of the thermogram of the precipitate.

Ignition is done in a silica or porcelain crucible directly on a bunsen flame. Drying is done in a sintered crucible using an air oven.

It is ignited / dried, cooled and weighed till we get constant weight.

Sequestering Agents :

They are reagents added to eliminate the interference by a substance in estimation. These are also called masking agents.

Explanation :

If we want to estimate Mg^{2+} gravimetrically in presence of Cu^{2+} using oxine, then we have to eliminate Cu^{2+} . For this we use CN^- which forms a soluble complex with Cu^{2+} as shown below ;



Thus Cu^{2+} is prevented from reacting with oxine. Now CN^- is the sequestering agent.

The process of eliminating an ion or substance using a chemical agent so that it does not interfere in the estimation of another ion or substance is called sequestration.

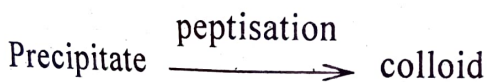
Uses :

1. Mg^{2+} can be estimated gravimetrically with oxine in presence of Cu^{2+} Using CN^- as the sequestering / masking agent.
2. Ca^{2+} can be estimated with suitable reagent in presence of Cu^{2+} using cyanide ion as sequestering agent.
3. In the presence of EDTA, beryllium may be precipitated with ammonia in presence of chromium, cobalt, cadmium, iron, copper, lead, manganese, zinc, aluminium, bismuth etc.

4. Uranium can be separated from numerous other ions by precipitation with oxine from an EDTA solution at pH 5.3.
5. Copper has been masked with EDTA in the polarographic determination of antimony in alloys.
6. Precipitate of soaps as their calcium and magnesium salts by hard water can be checked by transforming these metals into soluble complexes of EDTA.

Peptisation :

Peptisation is a process by which a precipitate is converted into a colloid.



This can be done in two ways

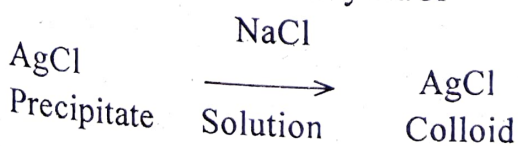
- i. By adding suitable ions
- ii. By adding the dispersion medium.

The substance added for this purpose is called the peptising or dispersing agent.

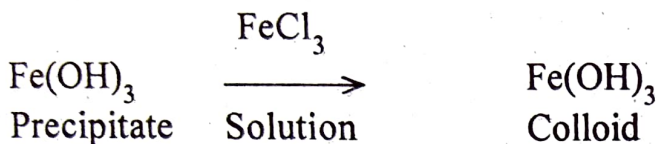
Peptisation by ions :

In this method a small concentration of an electrolyte is added to a precipitate in presence of the dispersion medium. The ions present in the electrolyte act as peptising agent. We get a colloid. In this process the precipitate particles adsorb suitable common ions from the added electrolyte. Their boundary potentials are raised. So they are dispersed into the medium as a result of the electrostatic repulsion of the adsorbed ions.

E.g. 1 : Freshly precipitated silver chloride is peptised by a solution containing chloride ions. Say NaCl



E.g. 2 : When freshly prepared ferric hydroxide precipitate is washed with small quantities of ferric chloride solution, ferric hydroxide colloids is obtained.



Peptisation by dispersion medium :

In this method a precipitate is converted into a sol by simply shaking it with the dispersion medium. By this method we can prepare lyophilic sols. E.g, Gelatin is peptised by water.

Uses :

Thus peptisation is used for preparing both lyophobic and lyophilic colloids.

Crucibles

Simple crucibles :

Simple crucibles serve only as containers. Porcelain, aluminum oxide, silver and platinum crucibles maintain constant mass within the limits experimental errors. Simple crucibles of Ni, Fe, Mg and Au are used as containers for the high temperature fusion of samples that are not soluble in aqueous reagents. Attack by both the atmosphere and the contents may cause the crucibles to suffer mass changes.

Filtering crucibles

Filtering crucibles serve not only as container but also as filters. A vacuum is used to hasten the filtration. A tight seal between crucible and filtering flask is accomplished with any of several types of rubber adaptors. Collection of a precipitate with a filtering crucibles is frequently less time consuming than with paper.

Calculation in gravimetric analysis

Example :

Weighing of Barium Sulphate Precipitate

Calculation

Weight of BaSO ₄ present in 20 ml of the solution	=0.2440g
Weight of BaSO ₄ present in 100 ml of the solution	=0.2440 x 5
	= 1.2200g
233.46g of Barium Sulphate contains	= 137.36g of barium
1.2200g of barium sulphate contains	= $\frac{122 \times 137.36}{233.46}$
	= 0.7178g of barium
Weight of Barium Sulphate present in the whole of the given solution	= 0.7178g

University Problems

1. In a gravimetric estimation of chloride as AgCl 20 ml of chloride solution gave 0.2866 g of precipitate. Calculate the amount of chloride in one litre of the solution.

Solution : One mole of AgCl \equiv One g. ion of Cl⁻
i.e., 107.87 + 35.45g AgCl = 35.45g of Cl⁻

$$0.2866 \text{ g of AgCl} = \frac{35.45}{143.32} \times 0.2866 \text{ g of Cl}^-$$

$$= 0.07089 \text{ g of Cl}^-$$

i.e., 20 ml of chloride solution contains 0.07089 g of Cl⁻

$$1000 \text{ ml contains} = \frac{0.07087}{20} \times 1000$$

$$= 3.545 \text{ g of Cl}^-$$

Answer : Amount of chloride in one litre of the solution = 3.545g

2. In a gravimetric estimation of sulphate as barium sulphate 20 ml of sulphate solution give 0.1267 g of precipitate. Calculate the amount of sulphate per litre of the solution.

Solution : One mole of $\text{BaSO}_4 = 1 \text{ g ion of SO}_4^{2-}$

$$\text{i.e., } 137.34 + 32.06 (16 \times 4) \text{ g of BaSO}_4 = 32.06 + (16 \times 4) \text{ g of SO}_4^{2-}$$

$$\text{i.e., } 233.4 \text{ g of BaSO}_4 = 96.06 \text{ g of SO}_4^{2-}$$

$$0.1267 \text{ g of BaSO}_4 = \frac{96.06}{233.4} \times 0.1267$$

$$= 0.05215 \text{ g of sulphate}$$

i.e., 20 ml of sulphate solution contains 0.05215 g of sulphate.

$$1000 \text{ ml contains} = \frac{0.05215}{20} \times 1000 = 2.607 \text{ g}$$

Answer : The amount of sulphate per litre of the solution = 2.607 g

University Questions

1. Describe the principle of gravimetric analysis with an example.
2. Explain the term / what do you mean by gravimetric factor (with the help of two examples).
3. Write a note on the choice of precipitants.
4. Write a note on specific precipitants with examples.
5. Draw the structure of Oxine. What are its properties and analytical uses?
6. Write down the structure of Cupron.
7. Draw the structure of cupferron. What are its properties and analytical uses?
8. Write a note on selective precipitants with examples.
9. What are the two types of organic precipitants?