Last Chapter ⑧ Gravimetric Analysis

Date 08/6/22
Page 1

Gravimetric Analysis

Gravimetry analysis or quantitative analysis by weight is the process of isolating and weighing an element or definite compound.

Ex:- Progesteron injection,

The content of progesteron ~~injection~~ quantitatively determine by this method In other option when other component in a sample are difficult to separate by physical method chemical reaction is employed to convert the substance to be analysed to chemically equivalent amount of some other substance which can be ~~A~~ separated, purified and weigh.

Advantage:

① It is accurate and precise when using analytical balances

(ii) Positive sources of errors are readily checked, since filtrate can be tested for completeness of the precipitation and precipitate may be examined for the presence of impurities.

(iii) It is absolute method.

(iv) Relative ~~and~~ ~~extensive~~ inexpensive apparatus is required.

★ Disadvantage:

i) Measure disadvantage is that the procedure is time consuming.

ii) It consist of following steps:

i) Precipitation
ii) Digestion
iii) Filtration
iv) Washing of Dried ppt.
v) Drying and ignition of ppt.
vi) Weighing of ppt.

Q. Washing and drying of ppt

Gravimetric consists of following steps:

1. Precipitation :

It is an important step in which the anion or cation to be determined is precipitated from a sol^n of the substance in the form of insoluble compound consisting of either difinite composition itself or leaves a residue of definite composition on ignition.
For precipitation, a slight excess of precipitating agent is added to achieve complete precipitation.

→ Precipitation step is the most critical step for successfull gravimetric analysis. The desire constituent which is to be analyzed, must be precipitated quantitatively. The precipitate must be pure.

2. Digestion

To increase the coarseness of the precipitate for easy filteration, the contents are either left for sometimes to settle or they are boiled. The procedure is termed as digestion of the precipitate.

3. Filtration

These are two methods for separating a precipitate from the mother liquor for the purpose of drying or ignition to constant weight. suspension of solid is filtered through the filter paper or other means such as Gooch and Sintered silica crucible. Gooch crucible is particularly suitable where traces of precipitate can never be separated from ordinary paper,

Sintered glass crucible have porous bottom of Growl glass. Sintered glass disk is available in various porosity grade ~~ragging~~ ranging from 0 to 5. The 0 is maximum pore size of 150 to 250 micrometer while, 5 size has pore size of 1-2 micrometer It is used for bacteriological filteration Grade 3 is used but fine precipitate such as barium sulphate grade 4 is used.

4). Washing of dried ppt.

Washing . Most ppt are produced in presence of one or more soluble compounds. Since the soluble compounds are frequently non-volatile at the drying temperature of the ppt, it is necessary to wash the ppt to remove impurities as completely as possible. The ideal washing solution should comply following conditions:-

① It should have not solvent action on the ppt but dissolve foreign substance easily. ~~should~~

② It should have no dispersive action on a ppt

③ It should contain no substance which is likely to interfare with substituent determination in the filterate.

④ It should be easily volatile at the drying temp of the ppt.

⑤ It should not form any volatile or insoluble product with the ppt.

⑤ Drying and ignition of ppt.

To remove the extra amount of water drying is required after washing...... drying of ppt is done by using whatmann filter paper dried in an oven at about 110°c - 120°c. ~~aft~~ After drying the crucible is allow to cool in a dessicator and then ~~bake~~ weigh with the ppt. Heating, colling and bweighing is repitate till Constant ~~wet~~ wt is obtained.

Ignition

The ignition of ppt Can be described under following two conditions

① Ignition and the incarnation of filter paper. The filter paper having ppt is taken out of the funnel carefully and open in such a way that the fingers don't touch the ppt. Alternatively the ppt. may also be collected over a clean and dried glazed paper. ~~with~~ Take the help of ~~figure~~ feather or camel brush to collect the fine particles of the ppt. Now cover the ppt with a funnel and keep it safe.

Fold the filter paper several times so that it looks like a long small cone. Catch the top side of the cone with ~~the~~ a pair of tong by one hand and ignite the cone by means of a blaze by the burner in other hand urgently. horizontally.

Collect the ash in previously weighed crucible placed on a glazed paper. The crucible is then heated strongly to brush off all the carbon to a white ash.

~~Thee~~

During ignition The smart ignition of ppt may kept reduced by the carbon of the paper into a compound (or metal) altogether different ~~co~~ from the compound in which determination is ~~to~~ sought.

The ash is treated with suitable reagents to get back to form (compound) in which it is finally to be weigh. The step is called the ash treatment.

⑥ Weighing of ppt.

Crucible containing the ppt is weight and the wt of ppt may be ~~of~~ obtained as.

① weight of empty crucible = x grams.

② Weight of crucible + ppt = Y grams.

③ Weight of precipitate = (Y − x) = Z grams.

Mechanism ~~Magnism~~ of ppt.

The formation of ppt involves two steps.

① Nucleatation :→ It is a process in which a minimum number of atoms, ions, and molecules joined together to give a stable solid. It also involves the formation of nucleus. and is further classified into two types.

and is ~~ff~~ further classified into two types

Ⓐ Spontaneous :-

Some times it is possible in supersaturated sol^n ions which will joints together to form nucleus. It is called spontaneous nucleation and it will occur its own.

Ⓑ Induced :

If a small ~~mixt~~ crystal of solid is added to the supersaturated sol^n, it will act as a nucleus for growth of a crystal. It is called induced nucleation. and requires ~~are~~ a seed particle to get things started.

~~A~~ ② ~~B~~ Crystal growth

It is achieved after nucleation. It is absorbed in two cases.

Ⓐ Diffusion of ion to the surface of growing crystal

Ⓑ Diffusion deposition of these diffused ion on crystal surface. Once a nucleation site is formed the other ion are attracted to the site and will result in the formation of large filterable particles.

Impurities and purity of precipitate.

Purity of Precipitate

Co-Precipitation

Co-precipitation is a state in which impurities are precipitate with analyte. It is a major problem concerned to the purity of analyte. Co-precipitation result into excess mass production. This problem can be overcome by digestion process to achieve pure analyte.

There are four types of co-precipitation:

i) Surface adsorption
ii) Mixed - Crystal formation
iii) Occlusion.
iv) Mechanical entrapment.

Surface adsorption and mixed crystal formation are equilibrium processes, whereas occlusion and mechanical entrapment arise from the kinetics of crystal growth

Surface adsorption:

Surface adsorption is common sources of co-precipitation where contaminants or impurities are get adsorbed on the surface of precipitate, It is common co-precipitation source.

The co-precipitated contaminant on the coagulated colloid consists of the lattice ion originally absorbed on the surface before coagulation and the counter ion of opposite charge held in the film of solution immediately adjacent to the particle. The net effect of surface adsorption is the carrying down surface contaminant soluble form with precipitate.

The purity of many coagulated colloids is improved by digestion.

Mixed Crystal Formation.

In mixed-crystal formation, one of the ions gets replaced by another ion having same charge and size in the crystal class. For example, $MgKPO_4$ in $MgNH_4PO_4$, $SrSO_4$ in $BaSO_4$. This problem

Can be solved by using more selective precipitating agent or reprecipitation of analyte can resolved this problem.

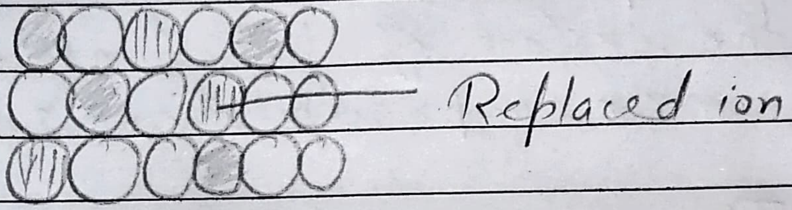

Fig → Mixed crystal formation.

Occlusion and Mechanical Entrapment.

Occlusion is a process where impurities are trapped within the pocket of crystal lattice. This process happens when rapid precipitation occurs.

Mechanical entrapment occurs when crystals lie close together during growth. Crystals grow together and in so doing trap a portion of the solution in a tiny pocket. Digestion process can solve this problem by forming large pure crystals. Digestion is often remarkably helpful in reducing these types of coprecipitation.

The rapid solution and reprecipitation that goes on at the elevated temperature of digestion opens up the pockets and allows the impurities to escape into the solution. If the precipitation rate is slow then occlusion and mechanical entrapment is minimum.

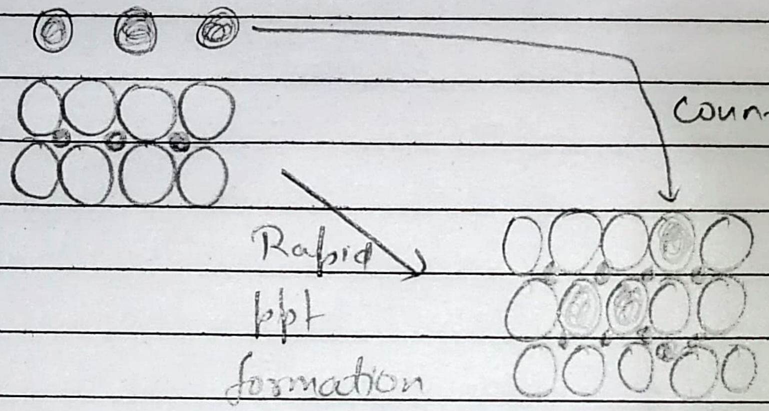

Fig → Occlusion.

Postprecipitation

Postprecipitation is the process by which impurities are deposited after the formation of desired ppt. When ppt. stands in contact with the mother liquor long period it becomes contaminated by an impurity on surface of the desired ppt. For ex: calcium oxalate precipitated as analyte and magnesium is also present in solution of oxalate then slowly magnesium oxalate gets, precipitated over desired calcium oxalate due to post-precipitation.