PHARMACEUTICAL ANALYSIS – BP102T

UNIT: III PRECIPITATION TITRATION, COMPLEXOMETRIC TITRATION, GRAVIMETRY AND DIAZOTISATION TITRATION

CLASS:27

TOPIC: GRAVIMETRIC ANALYSIS

Gravimetric analysis:

- **It** describes a set of methods used in analytical chemistry for the quantitative determination of an analyte (the ion being analyzed) based on its mass.
- The principleof this type of analysis is that once an ion's mass has been determined as a unique compound, that known measurement can then be used to determine the same analyte's mass in a mixture, as long as the relative quantities of the other constituents are known.
- The four main types of this method of analysis are *precipitation*, *volatilization*, *electro-analytical*,and *miscellaneous physical methods*.
- The methods involve changing the phase of the analyte to separate it in its pure form from the original mixture and are quantitative measurements.

STEPS INVOLVED IN GRAVIMETRIC ANALYSIS:

After the appropriate dissolution of the sample the following steps should be followed for a successfulgravimetric procedure:

1. **Preparation of the Solution**: This may involve several steps including adjustment of the pHof the solution in order for the precipitate to occur quantitatively and get a precipitate of desiredproperties, removing interferences, adjusting the volume of the sample to suit the amount of precipitating agent to be added.

2. **Precipitation**:

- This requires addition of a precipitating agent solution to the sample solution. Upon addition of the first drops of the precipitating agent, supersaturation occurs, then nucleation starts to occur where every few molecules of precipitate aggregate together forming anucleus.
- At this point, addition of extra precipitating agent will either form new nuclei or will build up on existing nuclei to give a precipitate.
- The Q is the concentration of reactants before precipitation, S is the solubility of precipitate inthe medium from which it is being precipitated.
- Therefore, to get particle growth instead of further nucleation we must make the relative supersaturation ratio as small as possible.
- The optimum conditions for precipitation which make the supersaturation low are:

a. Precipitation using dilute solutions to decrease Q

b. Slow addition of precipitating agent to keep Q as low as possible

c. Stirring the solution during addition of precipitating agent to avoidconcentration sites and keep Q low

d. Increase solubility by precipitation from hot solution

e. Adjust the pH to increase S, but not too much increase np as we do not want to lose precipitate by dissolution

f. Usually add a little excess of the precipitating agent for quantitative precipitation and check for completeness of the precipitation

3. **Digestion of the precipitate**:

- The precipitate is left hot (below boiling) for 30 min to one hour for the particles to be digested. Digestion involves dissolution of small particles and reprecipitation on larger ones resulting in particle growth and better precipitate characteristics. This process is called Ostwald ripening.
- An important advantage of digestion is observed for colloidal precipitates where

large amounts of adsorbed ions cover the huge area of the precipitate.

- Digestion forces the small colloidal particles to agglomerate which decreases their surface area and thus adsorption.
- Therefore, forming what is called a primary ion layer which attracts ions from solution forming asecondary or counter ion layer.
- Individual particles repel each other keeping the colloidal properties of the precipitate.
- Particle coagulation can be forced by either digestion or addition of a high concentration of a diverse ions strong electrolytic solution in order to shield the charges on colloidal particles and force agglomeration.
- Usually, coagulated particles return to the colloidal state if washed with water, a process called peptization.

4. **Washing and Filtering the Precipitate**: It is crucial to wash the precipitate thoroughly to remove all adsorbed species that would add to the weight of the precipitate. One should be careful nor to use too much water since part of the precipitate may be lost. Also, in case of

colloidal precipitates we should not use water as a washing solution since peptization would occur.

In such situations dilute nitric acid, ammonium nitrate, or dilute acetic acid may be used.

Usually, it is a good practice to check for the presence of precipitating agent in the filtrate of thefinal washing solution.

The presence of precipitating agent means that extra washing is required.

Filtration should be done in appropriate sized Gooch or ignition filter paper.

5. **Drying and Ignition**:

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

6. **Precipitation from Homogeneous Solution**:

To make Q minimum we can, in some situations, generate the precipitating agent in the precipitation medium rather than adding it.

For example, to precipitate iron as the hydroxide, we dissolve urea in the sample. Heating of the solution generates hydroxide ions from the hydrolysis of urea. Hydroxide ions are generated at all points in solution and thus there are no sites of concentration.

We can also adjust the rate of urea hydrolysis and thus control the hydroxide generation rate.

This type of procedure can be very advantageous in case of colloidal precipitates.