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STEPS INVOLVED IN GRAVIMETRIC ANALYSIS.

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STEPS

PREPARATION OF THE SOLUTION:

- **This may involve several steps including adjustment of the pH of the solution in order for the precipitate to occur quantitatively and get a precipitate of desired properties, removing interferences, adjusting the volume of the sample to suit the amount of precipitating agent to be added.**



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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 a nucleus. At this point, addition of extra precipitating agent will either form new nuclei or will build up on existing nuclei to give a precipitate.



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DIGESTION OF THE PRECIPITATE:

- **The precipitate is left hot (below boiling) for 30 min to 1 hour in order 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.**



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WASHING AND FILTERING THE PRECIPITATE:

- **It is crucial to wash the precipitate very well in order to remove all adsorbed species which will add to weight of precipitate. One should be careful not 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 the final 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**



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DRYING AND IGNITION:

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

PRECIPITATION FROM HOMOGENEOUS SOLUTION:

- **In order to make Q minimum we can, in some situations, generate the precipitating agent in the precipitation medium rather than adding it. For example, in order to precipitate iron as the hydroxide, we dissolve urea in the sample..**



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- **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.



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THANK YOU