

## GRAVIMETRIC TITRATIONS

Q. Write a short note on process of digestion/Ostwald ripening in gravimetry. OR What are advantages of digestion?

Ans. Digestion is the process in which the precipitate is allowed to stand in contact with mother liquor for about 12-24 hrs at room temperature or the mixture is warmed.

- 1) In this way complete precipitation takes place.
- 2) Bigger particles of precipitates are formed which can be easily filtered.
- 3) Smaller particles of precipitates goes into the solution and then they are redeposited on bigger particles which are easy to filter.
- 4) Co-precipitation is minimized; because it takes place in smaller particles. Therefore, it reduces co-precipitation and increases the size of particles, which are easy to filter.

Q. write a note on washing of precipitates. OR

What are the properties of ideal washing solution? Classify wash solutions.

Ans. The main objective of washing the precipitates is to remove the surface impurities from the precipitates as completely as possible. The composition of wash liquid selected depends on

- A) solubility and chemical properties of precipitates.
- B) Tendency of precipitate to undergo peptization.
- C) Impurities to be removed.
- D) Influence of traces of wash liquids upon precipitate before weighing.

Pure water is generally not used for washing, because it produces partial peptization of precipitate i.e. the coagulated particles go back into the colloidal state. Therefore, generally solution of some electrolyte is used, that possess common ion with that of the precipitate to reduce the solubility errors and should be easily volatilized.

Eg:  $\text{NH}_4^+$  salts,  $\text{NH}_3$  solution, dil. Acids are commonly used for washing.

Wash solutions are divided into 3 classes:

1. **Solutions which prevent precipitate from becoming colloidal & passing through filter:**  
eg: dilute  $\text{NH}_4\text{NO}_3$  solution is used for washing of  $\text{Fe}(\text{OH})_3$  (or Hydrated  $\text{Fe}_2\text{O}_3$ ) & 1%  $\text{HNO}_3$  is used for  $\text{AgCl}$ . Such tendency of becoming colloidal is observed with gelatinous or flocculated precipitates but not with well defined crystalline precipitates.
2. **Solution which reduce solubility of precipitate:**
  - ∫ Wash solution contains a moderate concentration of compound with one ion in common with precipitate because substance tends to be less soluble in presence of slight excess of common ion.
  - ∫ Organic solvents can sometimes be used for washing precipitates. Eg. 100ml of water at  $25^\circ\text{C}$  will dissolve 0.7 mg of  $\text{CaC}_2\text{O}_4$ , but same volume of dilute  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  dissolves negligible amount of salt.
3. **Solutions which prevents hydrolysis of salts of weak acids & bases:**
  - ∫ If ppt. is salt of weak acid & is slightly soluble it will try to hydrolyse into soluble product base.
  - ∫ Therefore wash liquid must be basic and therefore  
 $\text{Mg}(\text{NH}_4)\text{PO}_4 \quad \text{HPO}_4^{2-} + \text{OH}^-$   
Therefore it must be washed with dilute  $\text{NH}_3$  solution.Solubility losses can be minimized by taking less washing liquid every time rather than washing ppt. with large quantities of wash liquid.

Q. Define Filtration. What are various mediums used for filtration? Describe briefly.

Ans. Filtration is defined as separation of precipitate from mother liquor, so that precipitate & filtering media is quantitatively free from filtering solution. Different mediums are employed.

- a. Filter paper
- b. Filter mats of purified asbestos (gooch crucibles) or of Platinum (munroe crucibles)
- c. Porous fritted plates of resistance glass (pyrex) (sintered glass filtering crucibles) (silica) or of porcelain.

**Filter papers:** Quantitative filter papers are employed with small ash content (during manufacturing such papers, washing with HCl & HF is done).

**Paper Size :** Circles of 7, 9, 11, 12.5 cm diameter are generally available. Ash of 11 cm diameter paper is not more than 0.0001g. Hardened filter papers are made by further treatment with acid that gives extremely small ash values. These are more resistant to acids & alkalis & are used for quantitative work.

Eg. Whatman qualitative filter paper .

Size of filter paper depends on bulk of precipitate & not on of liquid to be filtered. Folded filter paper is placed within 1-2cm of top of funnel to increase the surface area.

**Crucibles:** These are used to collect precipitate in which it can be directly weighed. Gooch crucible are made of porcelain, silica, & rarely platinum.

The base is pierced with no. of small holes. Holes are covered by pad of asbestos, produced by sucking a slurry of asbestos fibres in water through crucibles under reduced pressure.

**Sintered glass crucibles:** Made up of resistance glass, have porous disc of sintered ground glass fused into the body of crucible.

**Filter disc:** Available with varying porosities from 0 (coarsest) to 5 (finest)

Porosity	0	1	2	3	4	5
Pore dia. ( $\mu\text{m}$ )	200-250	100-120	40-50	20-30	5-10	1-2