Session-2018-19

ऋते ज्ञानान्न मुक्तिः

GLA
UNIVERSITY
MATHURA
Recognised by UGC Under Section 2(f)

Accredited with A Grade by NAAC

Module-1

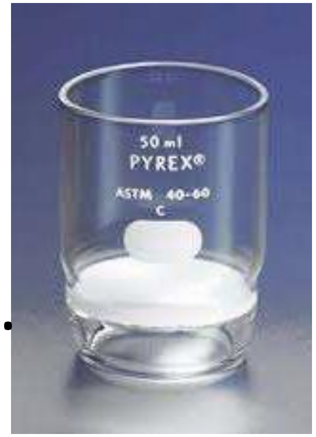
Theory of Volumetric and Gravimetric Analysis

Presented by:

Dr. Pankaj Kumar Rastogi

Assistant Professor

4. Filtration



- Ppt is separated from mother liquor.
- Choice depends on nature of ppt, cost of media and heating temp required for drying.
- Filtration medium used are
- – Filter papers
- – Crucible fitted with porous plate (Sintered glass filters)
- – Crucible to be used at high temperature

- Sintered glass crucibles are used to filter the precipitates.

5. Washing

- Co precipitated impurities especially those on surface, removed by washing the precipitate.

- Wet precipitate with mother liquor and which will also be remove by washing.

- Need to add electrolyte to the wash liquid because some precipitate cannot be wash with pure water, peptization occur.

- Eg HNO_3 for AgCl precipitate

6) Drying or ignition

- To remove solvent and wash electrolytes.
- Done by heating at 110 to 120°C for 1 to 2 hrs.
- May used high temp if precipitate must be converted to a more suitable form before weighing.
- Example: $MgNH_4PO_4$ convert to pyrophosphate $Mg_2P_2O_7$ by heating at 900°C.

7) Weighing

- After the precipitate is allowed to cool (preferably in a desiccator to keep it from absorbing moisture), it is weighed (in the crucible).

- Properly calibrated analytical balance

- Good weighing technique

Properties of precipitate

- The precipitate should be so insoluble that no significant loss occurs during filtration and washing.

- Physical nature of ppt should be such that it can be easily separated by filtration.

- The PPT should be stable to atmospheric condition.
- Have large crystals (Easier to filter large crystals)
- Be free of **contaminants.**

Impurities in precipitates

- When the precipitates is separated out from solution, it is always not preferably pure and may be contaminated even after washing.
- The amount of impurities depends on nature of precipitates and condition of precipitation.
- We must remove these impurities before determining the precipitate’s mass.

Impurities in precipitates

- It may be due to:
- Co-precipitation: Co-precipitation is the phenomenon in which soluble compounds are removed from solution during precipitate formation.

- It is the precipitation of an unwanted species along with your analyte of interest; occurs to some degree in every gravimetric analysis;

- It cannot be avoided, but can be minimized by careful precipitation and a thorough washing of the precipitate.

- Co-precipitation can be happen by:

❖ **Surface adsorption**

❖ **Mixed crystal formation**

❖ **Occlusion and**

❖ **Mechanical Entrapment**

Surface adsorption

Surface adsorption is a common source of co-precipitation that is likely to cause significant contamination of precipitates with large specific surface areas.

- Unwanted material is adsorbed onto the surface of the precipitate.
- Digestion of a precipitate reduces the relative surface area and, therefore, the are available for adsorption of impurities.
- Washing can remove impurities bound to the surface.

Mixed crystal formation

- In mixed-crystal formation, one of the ions in the crystal lattice of a solid is replaced by an ion of another element.

- For this exchange to occur, it is necessary that the two ions have the same charge and that their sizes differ by no more than about 5%.

- Furthermore, the two salts must belong to the same crystal class. For example, $MgKPO_4$ in

$MgNH_4PO_4$, $SrSO_4$ in $BaSO_4$, and MnS in CdS.

- The extent of mixed-crystal contamination increases as the ratio of contaminant to analyte concentration increases.

- Mixed-crystal formation is troublesome because little can be done about it.

- Separation of the interfering ion may have to be carried out before the final precipitation step.

- Alternatively, a different precipitating reagent may be used.