Quadrant II – Transcript and Related Materials

Programme: Bachelor of Science (Third year)

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Paper Title: Basic Topics in Analytical Chemistry

Unit: 02

Unit Title: Quantitative Analysis

Module Name: Gravimetric Analysis: Post Precipitation and Digestion

Module No: 14

Name of the Presenter: Ms. Priyanka R. Fernandes

Notes

❖ Post Precipitation

- Post precipitation is the precipitation of impurity after some time of the sample precipitation. It refers to the precipitation which occurs on the surface of the first precipitate after its formation.
- This process occurs with sparingly soluble substances which form super saturated solutions and have an ion in common with the primary precipitate.
- Post precipitation increases with increase in time.

Mechanism:

1. Analyte ion will precipitate out first due to precipitating agent.
2. After this, other ions are precipitated by the same precipitating agent.
3. Precipitation of these other ions is known as post precipitation.

Example: When Mg oxalate is present in Ca oxalate solution.

Oxalate ions form primary layer and Mg ions form secondary layer.

Thus, in the precipitation of Calcium oxalate in the presence of Magnesium, magnesium oxalate separates/precipitates out gradually upon the calcium oxalate.

- Rate of reaction is slower in post precipitation and it occurs generally during digestion process.
- In the presence of the primary precipitate, the particles of the primary precipitate act as nuclei for the post precipitation of the impurity from its supersaturated solution.
- The longer the precipitate is allowed to remain in contact with the solution, greater will be the error (contamination) caused due to this.
- Post precipitation always gives positive error.

Prevention of Post Precipitation:

Precipitate can be filtered quickly.

Digestion

Reprecipitation

Glass piece can be added to act as nucleus for the impurities

Post precipitation differs from coprecipitation in the following aspects:

a) The contamination increases with the time that the precipitate is left in contact with the mother liquor in post- precipitation but decreases in co-precipitation.
b) With post precipitation, contamination increases the faster the solution is agitated either mechanically or thermally. With coprecipitation, agitation decreases the contamination.
c) Magnitude of contamination by post precipitation may be much greater than by co-precipitation.

Post Precipitation	Co-Precipitation
Contamination increases with time that the precipitate is left in contact with the mother liquor.	Contamination decreases with time.
Contamination increases with increase in the rate of agitation either mechanically or thermally.	Increase in the rate of agitation decreases the contamination.
Magnitude of contamination is much greater.	Contamination is less in case of co-precipitation.

❖ DIGESTION of Precipitates

The objective of digestion is to improve the filterability of the precipitate and its purity. This ensures complete precipitation in a form which can be readily filtered.

Digestion decreases the no. of colloidal particles.

This process is also known as the 'Ostwald Ripening' of the precipitate as it promotes a significant increase in average particle size of the precipitate.

PROCESS

Usually, colloidal particles (10-2000 Å diameter) of the precipitate pass through the pores of the filter paper and are not separated from the supernatant.

The process of digestion is usually carried out by allowing the precipitate to remain in contact with the mother liquor usually at elevated temperatures, for some time before filtration.

During the digestion process, the smaller particles which exhibit higher solubility tend to dissolve, making the solution super saturated with respect to the larger ones.

To restore the solution equilibrium, the dissolved material deposits on the larger particles i.e. the larger particles grow at the expense of the smaller ones.

Two different methods can be employed during Digestion:

Method 1: Liquid is heated and cooled. On cooling, colloidal particles adsorb on the nuclei of the precipitate.

OR

Method 2: Liquid is kept for 12-24 hours. Colloidal particles are adsorbed on surface of precipitate.

Effects of Digestion:

The process of digestion is extremely crucial especially for crystalline precipitates.

During digestion, imperfections in the crystals tend to disappear and absorbed impurities tend to go back into the solution.

This may be attributed to the crystals of irregular shape becoming more regular in shape and denser, thus resulting in a decrease in surface area and reduction in extent of adsorption.

NOTE:
Digestion for gelatinous precipitate should be carried out for longer period to permit coagulation before filtration.