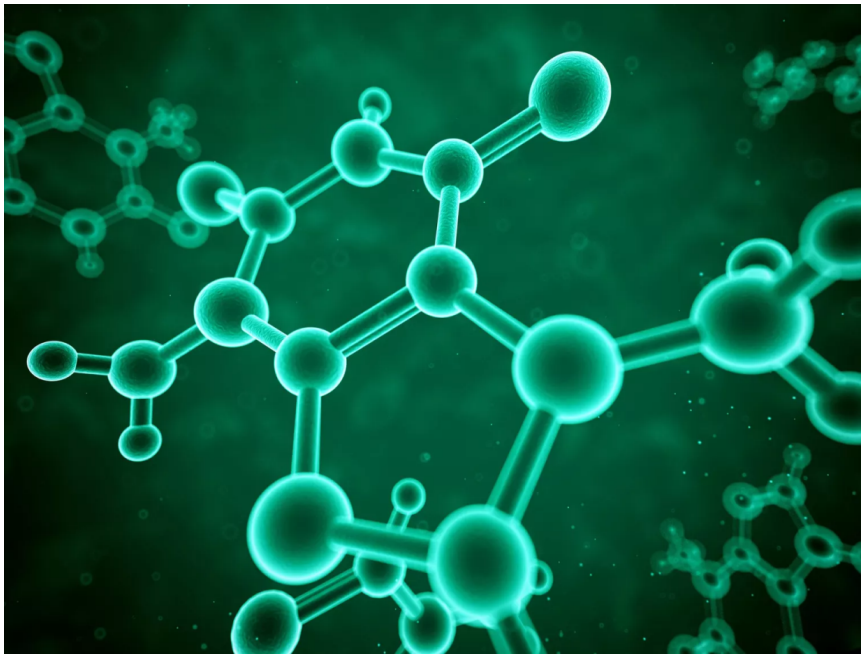
Analytical Chemistry



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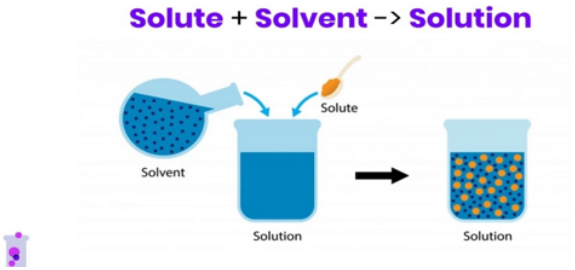
Lecture 3

- **Step of gravimetric analysis:**

1. **<u>Dissolve the analyte</u>**

If the analyte is solid weight as your procedure then dissolve it after choosing the suitable solvent or else take a suitable volume from the liquid analyte.



Purity of solvent : the solvent must be <u>free from impurities</u> that could interfere with the analysis.

Temperature and Time : these need to be optimized to ensure efficient dissolution of the sample with out causing unwanted reaction.

Amount of sample : ensure that the correct mass of the sample is dissolved to maintain accuracy in the subsequent analysis.

- The primary purpose of dissolving a sample in gravimetric analysis is to convert the analyte into a soluble form for further reaction.
- Ensured the sample must be completely dissolved in the solvent before proceeding with the precipitation step in gravimetric analysis.

When dissolving a sample for gravimetric analysis the solvent must be inert and not react with the analyte.

- If the sample does not dissolve completely during the dissolution step added more solvent and heat the mixture.
- heat and stirring to increase the solid solubility (form of homogeneous solution).
- Presence of impurities in the solvent could potentially interfere with the dissolution process of a sample.
- the typical role of heating during the dissolution of a sample to increase the rate of dissolution of the sample.
- Hydrochloric acid solvents is commonly used for dissolving metal oxides in gravimetric analysis.

2. First treatment of the analyte :

- After the sample was dissolved we should initialize the reaction medium for the reaction between the analyte and precipitating agents and we should conceder these point:
  - volume of solution (50 -200 ml)
  - solution temperature
  - PH of the solution
  - removal of interferences

3. Precipitation :

- Precipitation process involves the conversion of analyte quantitatively in to sparingly soluble substance called precipitate on the addition of chemical reagent called precipitating agent.

- **Properties of precipitating agent used in gravimetric analysis** :

➢ the ideal **gravimetric precipitating agent** should react specifically or at least selectively with the analyte.

➢ Specific reagents, which are rare, react only one with a single chemical species.

➢ Selective reagents, which are more common , react with a limited number of species.

➢ In addition to specificity and selectivity the ideal precipitating reagent would react with the analyte to give a product that is :

- Enough particle **size** for retaining on filter
- **High purity** (free of contaminant)
- **Low solubility** that no significant loss of the analyte occurs during filtration and washing
- Unreactive with air (stable)
- **Known chemical** structure after it is dried or if necessary ignited.

4. Degestion

- The precipetate 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 reprecipetation on large ones resulting in particles growth and better precipetate charactrastcs
- The primary purpose of digestion in gravimetric analysis to allow the precipitate to grow into larger crystals and reduce co-precipitation.
- The effect of digestion on the purity of the precipitate It increases the purity of the precipitate by allowing small particles to aggregate into larger crystals.
- Typically used to promote digestion in gravimetric analysis heating the solution or allowing it to stand for a prolonged period.

5. Filtration

- Filtration in gravimetric analysis is a critical step used to separate the solid precipitate from the liquid (filtrate).
- The goal is to isolate the analyte (as a precipitate) from any remaining solution, while minimizing the loss of the analyte and preventing contamination of the sample.

- The filtration process typically involves using filter paper and a filtration apparatus (e.g., funnel, Buchner funnel, or filter flask).

6. Washing

Washing of the sample in gravimetric analysis refers to the process of removing impurities.

excess reagents, or soluble substances from the precipitate after it has been separated from the solution through filtration.

This step is essential to ensure that the precipitate is pure and only contains the analyte of interest, leading to more accurate and reliable results.

Distilled water is commonly solvent used for washing the precipitate in gravimetric analysis.

Distilled water is neutral and does not contain ions that could contaminate the precipitate.

7. Drying and burning

- The precipitate is dried to remove water and other volatile substances from the precipitate
- Drying ensures that the mass measured after drying is solely that of the solid precipitate, not including any moisture.
- In gravimetric analysis Temperature is drying of the precipitate typically done at 100-110Â°C.

- Burning refers to heating the precipitate at a much higher temperature, often in a furnace or crucible, to remove any remaining volatile substances (e.g., organic matter) and achieve a constant weight.
- Temperature is burning (ignition) of the precipitate typically performed at 500-800Â°C.