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Gravimetry Analysis

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Gravimetry Analysis

Gravimetric Analysis is a quantitative method in analytical chemistry used to determine the amount of an analyte based on the mass of a solid. It involves converting the analyte into a pure, stable, and weighable form.

Principle of Gravimetric Analysis:

Gravimetric analysis is based on the **law of conservation of mass**. The amount of the desired constituent (analyte) present in a compound is determined by converting it into a pure and stable compound of known composition that can be weighed accurately.

- The most common gravimetric method involves **precipitation**: the analyte is precipitated out as an **insoluble compound**, which is filtered, washed, dried (or ignited), and weighed.

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Steps Involved in Gravimetric Analysis

S.No	Step	Description
1	**Preparation of Solution**	Dissolve the sample containing the analyte in an appropriate solvent, usually water or acid.
2	**Precipitation**	Add a suitable reagent to form an insoluble precipitate with the analyte. Conditions like temperature, pH, and reagent concentration are controlled to ensure complete and pure precipitation.
3	**Digestion of Precipitate**	The precipitate is allowed to stand (age) to improve filterability and purity by forming larger, purer crystals.
4	**Filtration**	The precipitate is separated from the solution using filter paper or a crucible.
5	**Washing**	The precipitate is washed with distilled water or a suitable solvent to remove impurities (e.g., adsorbed ions).
6	**Drying/Ignition**	The precipitate is dried in an oven or ignited in a furnace to convert it into a known and stable chemical form.
7	**Weighing**	The final stable form of the precipitate is weighed using an analytical balance.
8	**Calculation**	The amount of analyte is calculated from the mass of the dried precipitate using stoichiometry.

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Co-precipitation

and

Post-precipitation

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Co-precipitation

Definition:- Co-precipitation is the phenomenon where **impurities that are normally soluble** in the solution **get incorporated** into the precipitate along with the analyte during its precipitation.

Types of Co-precipitation:

Type	Description	Example
Surface Adsorption	Impurities adhere to the surface of the precipitate, especially in colloidal particles.	Adsorption of alkali metal ions on $BaSO_4$ precipitate.
Occlusion	Impurities get trapped within the crystal as it grows rapidly.	Na^+ or Cl^- trapped inside AgCl crystals.
Inclusion (Isomorphous replacement)	Foreign ions of similar size and charge replace the main ion in the crystal structure.	K^+ replacing NH_4^+ in NH_4MgPO_4.

How to Minimize Co-precipitation:

1. Use **dilute solutions**
2. **Slow addition** of precipitating reagent
3. **Stirring** the solution
4. **Digesting** the precipitate (aging it in solution)
5. **Re-precipitation** (dissolving and precipitating again)

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Post-precipitation

Definition:

Post-precipitation occurs when **impurities precipitate after the main precipitation is complete** and deposit on the already-formed crystals of the desired compound.

- The impurity forms **its own solid** and physically deposits on the main precipitate.
- It usually happens **if the precipitate is left standing too long** in the solution.

How to Prevent Post-precipitation:

1. **Filter the precipitate immediately** after digestion
2. **Avoid prolonged standing** of the precipitate in the mother liquor
3. Use **selective reagents** to prevent formation of other precipitates

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Estimation of barium sulphate.

Link:- https://pharmrecord.com/bp108p/

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Diazotisation Titration

Definition:- **Diazotisation Titration** is a type of **volumetric titration** used to estimate **primary aromatic amines**, based on their reaction with **nitrous acid** to form **diazonium salts** under **cold, acidic conditions**.

- It is commonly used in **pharmaceutical and dye analysis**, especially for the **quantitative determination of sulfa drugs**.

Principle of Diazotisation Titration

Principle:-In diazotisation titration, a **primary aromatic amine** reacts with **nitrous acid (HNO_2)** under cold and acidic conditions to form a **diazonium salt**.

Since **nitrous acid is unstable**, it is generated **in situ** by reacting **sodium nitrite ($NaNO_2$)** with **hydrochloric acid (HCl)**.

Stepwise Chemical Reactions:

Generation of Nitrous Acid (in situ):- $NaNO_2 + HCl \rightarrow HNO_2 + NaCl$

Diazotisation Reaction (Main Reaction):- $Ar\text{-}NH_2 + HNO_2 + HCl \rightarrow Ar\text{-}N_2^+Cl^- + 2H_2O$

Where:

- **$Ar\text{-}NH_2$** = Primary aromatic amine (e.g., aniline)
- **HNO_2** = Nitrous acid (generated in situ)
- **$Ar\text{-}N_2^+Cl^-$** = Diazonium chloride (diazonium salt)

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Methods of Diazotisation Titration

S.No	Method	Description
1	**Direct Titration Method**	❑ The sample containing a primary aromatic amine is directly titrated with standard sodium nitrite ($NaNO_2$) solution at 0–5°C in an acidic medium (usually HCl). ❑ The endpoint is detected using starch-iodide paper (external indicator) or KI-starch solution (internal indicator), which gives a blue color due to liberated iodine.
2	**Back Titration Method**	❑ An excess known amount of standard sodium nitrite is added to the amine solution. ❑ After complete diazotisation, the unreacted nitrite is titrated with a standard sulphanilic acid solution or another standard aromatic amine. ❑ This method is useful when the endpoint is difficult to detect directly.
3	**Blank Titration Method**	❑ A blank titration is performed under the same conditions but without the analyte (amine). ❑ It helps determine how much nitrite reacts with the acid alone. ❑ The volume consumed in the blank is subtracted from the main titration to get the accurate volume of nitrite that reacted with the analyte.

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Applications of Diazotisation Titration

S.No.	Application Area	Details / Examples
1	**Pharmaceutical Analysis**	Quantitative estimation of sulfa drugs like sulfanilamide, sulfadiazine, sulfapyridine
2	**Dye Industry**	Analysis of primary aromatic amines used in azo dye manufacturing
3	**Quality Control**	Determination of purity of raw materials containing aromatic amines
4	**Organic Chemistry Research**	Identification and estimation of primary aromatic amines in synthesized compounds
5	**Forensic Chemistry**	Detection and analysis of aromatic amines in criminal or toxicological samples
6	**Environmental Testing**	Monitoring of industrial effluents containing aromatic amines
7	**Analytical Method Development**	Standardization of sodium nitrite solution and indicator systems

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Thank you

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