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PHARMACEUTICAL ANALYSIS I

UNIT 4

TOPIC :

- **Redox titrations**
 - (a) Concepts of oxidation and reduction
 - (b) Types of redox titrations (Principles and applications)
Cerimetry, Iodimetry, Iodometry, Bromatometry, Dichrometry,
Titration with potassium iodate

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Redox Titration

- In titration the redox/ oxidation- reduction reaction are more extensively used for analysis as compared to the precipitation reaction and acid base reaction
- Reduction is defined as the gain of one or more electron by atomic species or molecules
- Oxidation is loss of one or more electron by the atomic species or molecules oxidation is also known as de electronation
- A titration which deals with a reaction involving oxidation and reduction of certain chemicals species are known as redox Titration

Agents

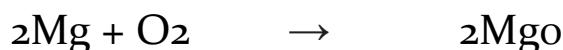
★ **Reducing agent** : Sodium thiosulphate, ferrous sulphate, titanous sulphate, oxalic acid are some of the reducing agents.

★ **Oxidising agents**: Potassium dichromate, potassium bromate etc are some of the oxidising agents.

Concepts of oxidation and reduction

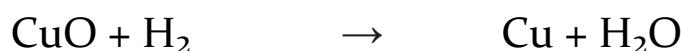
Oxidation

- ❖ Oxidation is the loss of electrons or an increase in the oxidation state of an atom, an ion, or of certain atoms in a molecule.
- ❖ Example: The reaction between magnesium metal and oxygen to form magnesium oxide involves the oxidation of magnesium.



Reduction

- ❖ Reduction is the gain of electrons or a decrease in the oxidation state of an atom, an ion, or of certain atoms in a molecule.
- ❖ Example: Copper oxide can be reduced to copper by heating it while passing hydrogen gas over it. The hydrogen gets oxidised to form water



Types of redox titrations (Principles and applications)

- ✚ Cerimetry
- ✚ Iodimetry
- ✚ Iodometry
- ✚ Bromatometry
- ✚ Dichrometry
- ✚ Titration with potassium iodate

Cerimetry

Principle:

- Oxidation-reduction titrations involving cerium sulphate as an oxidizing agent is called as ceriometry titration.
- Cerium sulphate is powerful oxidant and can be used only in acidic solution.
- In natural solution ceric hydroxide (hydrous ceric oxide) or basic salts precipitate.
- Cerium salts have intense yellow color and end point detection can be possible without any indicator in hot solution

Advantages:

- ✓ Cerium (IV) solutions are stable over prolonged period. They need not be protected from light, and may be even boiled for a short time without appreciable change in concentration.
- ✓ Cerium sulphate can be used to determine the reducing agents in presence of high concentration of HCl. This an advantage over KMnO_4
- ✓ Cerium (IV) solutions in 0.1M solutions are not too highly colored to obstruct vision when reading meniscus in burettes.

- ✓ In reaction of Cerium (IV) salts in acid solution with reducing agent, simple change takes place. Permanganate leads to several reduction products

Application:

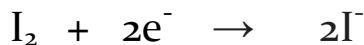
- Used in the assay of FeSO₄ (Ferrous sulphate).
- Used in assay of Ferrous fumarate tablets
- Used in assay of TiO₂ (Titanium dioxide)
- Used in assay of ascorbic acid tablets
- Used in assay of Paracetamol

Iodometry

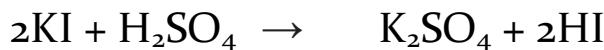
- Titration in which a standard iodine solution is used (Direct iodometric)

Principle:

- Iodometry covers titration with a standard solution of iodine. Iodometry deals with the titration of iodine liberated in chemical reaction. This method is based upon the inter conversion of elemental I₂ and iodide ion.



- In iodometry, the formation of iodine takes place as a result of hydriodic acid (HI), with an oxidizing agent. The HI is obtained directly in the reaction flask by the action of dilute HCl or H₂SO₄ on a solution of KI



- Free iodine is liberated as a result of the oxidation of KI in acidic solution. The iodine liberated is titrated with standard solution of sodium thiosulphate. The free iodine is converted to I⁻ ion (iodide ion) with reducing agent.

- Thus, iodometry titration is used for the quantitative determination of oxidizing agent and reducing agent.
- Standard solution of sodium thiosulphate and iodine are used in this method with starch as indicator. With free iodine it gives blue color.

Applications:

- ✓ Assay of $\text{Na}_2\text{S}_2\text{O}_3$ (Sodium Hypo Sulphite) by iodometry.
- ✓ Assay of sodium meta bisulfide by Iodometry

Iodometry

→ The titrations in which the equivalent amount of I_2 is liberated from KI by the sample the liberated I_2 is titrated against standard $\text{Na}_2\text{S}_2\text{O}_3$ solution such a type of indirect determination of strong oxidizing agents is called iodometry.

Principle:

- The titrations in which the liberated I_2 is titrated with a standard solution of $\text{Na}_2\text{S}_2\text{O}_3$ solution using starch-mucilage as indicator is called iodometric titrations. The indirect iodometric titration method is termed as iodometry.



Application:

- ✓ Assay of CuSO_4 (Copper sulphate)
- ✓ Determination of chlorine in bleaching powder.
- ✓ Assay of Phenol
- ✓ Assay of Chloramine
- ✓ Detection of chlorate

Bromatometry

Principle:

- Potassium Bromate is a powerful oxidizing agent which is reduced smoothly to bromide in the presence of HCl and which is then oxidized to give free bromine by bromate (excess).



Application:

- ✓ Assay of Isoniazid
- ✓ Determination of Hydroxylamine
- ✓ Determination of As/Sb. (Arsenic / Antimony)

Dichrometry

Principle:

→ Dichromate titrations or titrations with potassium dichromate in acidic solution are based on the conversion of dichromate ion containing hexavalent chromium into trivalent chromium ions.

Application:

- ✓ Determine the % of Fe (Iron) in sample of iron wire
- ✓ Determine ferrous and ferric ions in a solution
- ✓ Determine ferric ion % in solution of ferric alum
- ✓ Determine the % of Fe as haematite.

Titration with potassium iodate

- Potassium Iodate is a powerful oxidizing agent under suitable conditions, it reacts quantitatively with both iodides and iodine, arsenide's and other reducing agents.
- Iodate titration can be performed in presence of alcohol, saturated organic acid and many other kinds of organic matter.

Principle:

- ❖ The reaction between potassium iodate and reducing agents such as iodide ions or As_2O_3 (Arsenic oxidearsenous trioxide) in solutions of moderate acidity (0.12-2M HCl) stops at the state when the iodate is reduced to iodine.

