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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

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.

$$2Mg + O2 \rightarrow 2Mgo$$

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

$$CuO + H_2 \rightarrow Cu + H_2O$$

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 cerci 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 FeSO, (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

Iodimetry

- Titration in which a standard Iodine solution is used (Direct Iodometric)

Principle:

- Iodimetry covers titration with a standard solution of iodine. Iodimetry deals with the titration of iodine liberated in chemical reaction. This method is based upon the inter conversion of elemental I_2 and iodide ion.

$$I_2 + 2e^- \rightarrow 2I^-$$

- In Iodimetry, the formation if iodine takes place as a result of hydriodic acid (HI), with a oxidizing agent. The HI is obtained directly in the reaction flask by the action of dilute HCl or H_2SO_4 on a solution of KI

$$2KI + H_2SO_4 \rightarrow K_2SO_4 + 2HI$$

- Free iodine is liberated as 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, lodimetry 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 Na,S,O, (Sodium Hypo Sulphite) by iodimetry.
- ✓ Assay of sodium meta bisulfide by Iodimetry

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 Na2S2O3 solution such a type of indirect determination of strong oxidizing agents is called iodometry.

Principle:

- The titrations in which the liberated 12 is titrated with a standard solution of $Na_2S_2O_3$ solution using starch-mucilage as indicator is called iodometric titrations. The indirect iodometric titration method is termed as iodometry.

$$Na_2S_2O_3 + I_2 \rightarrow Na_2S_2O_3 + 2NaI$$

Application:

- ✓ Assay of CuSO4 (Cooper 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).

$BrO_3^- + 6H^+ + 6e^- \rightarrow 3Br_2 + 3H_2O$

$KBrO_3 + 5Br + 6HCL \rightarrow 3Br_2 + 6KCL + 3H_2O$

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 a 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 (Arsenic oxidearsenous trioxide) in solutions of moderate acidity (0.12-2M HCl) stops at the state when the iodate is reduced to iodine.

$$KIO_3 + 5KI + 6HCl \rightarrow 3I_2 + 6KCl + 3H_2O$$

$$2KIO_3 + 5H_3AsO_3 + 2HCl \rightarrow I_2 + 5H_3ASO_4 + H_2O + 2KCl$$