PHARMACEUTICAL ANALYSIS

UNIT 4 NOTES

REDOX TITRATION

- OXIDATION
- REDUCTION
- TYPES OF REDOX TITRATION
- IODIMETRY
- IODOMETRY

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REDOX TITRATION

- Redox titration are those titration that are based on the Redox Reactions.
- Redox – Reduction + Oxidation.
- Redox reaction are those reactions in which both oxidation and reduction reaction takes place.
- We can also say Redox reaction are those in which transfer of electrons takes place between Analyte and Titrant.

Oxidation

Oxidation can be defined as three ways.

- Addition of Oxygen
- Loss of Hydrogen
- Loss of electrons

Examples :

- In terms of oxygen : $SO_2 + O \rightarrow SO_3$
- In terms of Hydrogen : $H_2S + Cl_2 \rightarrow S + 2HCl$
- In terms of electrons : $Na \rightarrow Na^+ + e^-$

- Oxidation can be remembered by LEO (loss of electrons)

Reduction

Reduction is just opposite to oxidation, it can be defined as :

- Removal of Oxygen
- Gain of Hydrogen
- Gain of Electrons

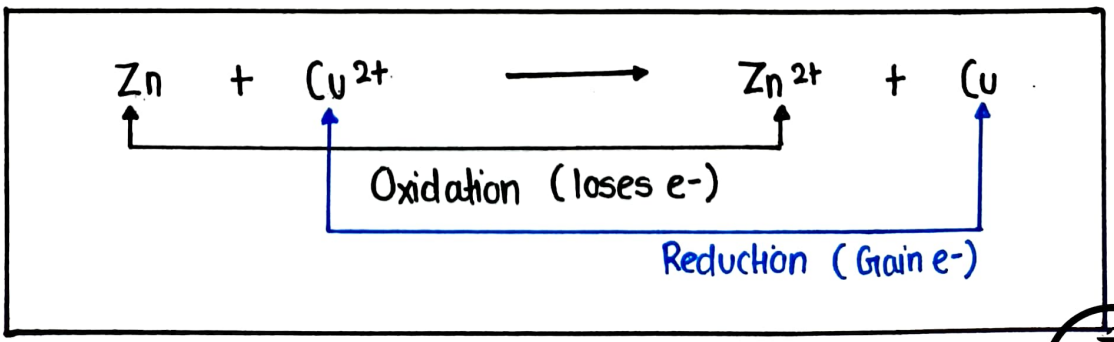
Example :

- In terms of oxygen : $CuO + 2H \longrightarrow Cu + H_2O$
- In terms of hydrogen : $C_2H_2 + 2H \longrightarrow C_2H_4$
- In terms of electrons : $Cl + e^- \longrightarrow Cl^-$

- Reduction can be remembered by GIEO (gain of electroons)

REDOX REACTIONS

- Redox reactions are those in which both oxidation and Reduction takes place.
- In redox reactions one substance loses or gives up the electrons and other substance receives the electrons
- The first substance is oxidized and other is reduced.

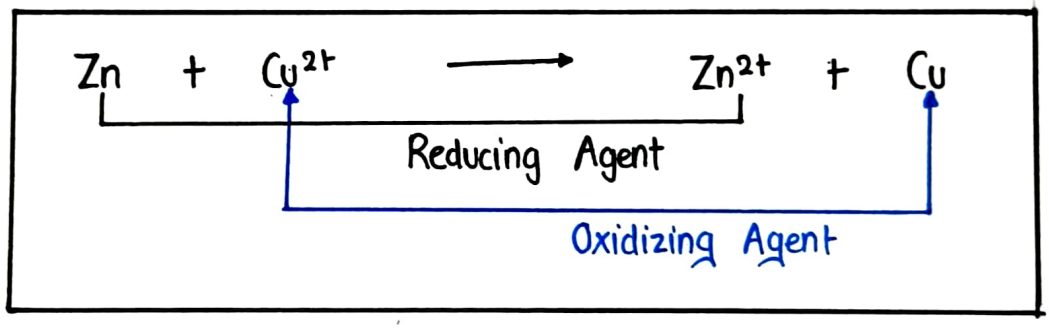


Oxidizing Agents

- Oxidizing Agents are those substances which do oxidation of others and itself gets reduced.
- We can simply say substance which gain electroons are called as Oxidizing Agents.

Reducing Agents

- Reducing Agents are those substances which do reduction of others and itself gets oxidized.
- We can simply say substance which loses or donates electrons are called as reducing agents.



REDOX INDICATORS

- Indicators that are used in Redox Titration are known as Redox Indicators.
- They are also known as Oxidation- Reduction Indicator.
- These are the substances that shows visible colour change at the end point of the redox titration.
- They shows different colours in their oxidized and reduced form.

Types of Redox Indicators

Redox indicators can be generally classified into three categories :

① Self Indicator
② External Indicator
③ Internal Indicator

Self Indicator

- Self Indicators are those in which titrant itself act as an indicator.
- In this we don't use any external indicator.
- Self Indicators are involved in the titration as titrant and at the end point they themselves changes colour and indicating the completion of reaction.
- example : $KMnO_4$ (potassium permanganate) is the best example of self Indicator, at the end point of the titration it shows pink colour.

External Indicator

- External Indicators as the name says, they are not added to the titration, they are kept outside.
- Titrated solution is taken and added dropwise time to time in the external indicator and the point at which it changes its colour simply determined by end point.
- example : Potassium Ferricyanide

Internal Indicators

- These are the actual Redox Indicators.
- These are the indicators which have different colours in their oxidized and reduced forms.
- They are added in the analyte solution and shows visible colour change at the end point.
- Internal indicators are generally colourless in reduced form.

INDICATOR NAME	OXIDIZED	REDUCED
Ferroin	Pale - Blue	Red
Diphenylamine	Violet / Blue	Colourless
Methylene Blue	Blue	Colourless
Starch - Iodine	Blue	Colourless
Nitroferroin	Pale - Blue	Red

TYPES OF REDOX TITRATION

Based on the Titrant used redox titration can be classified into following categories :

- Cerimetry
- Iodimetry
- Iodometry
- Bromatometry
- Dichrometry
- Titration with Potassium Iodate

CERIMETRY

Titrant : Ceroic Ammonium Sulphate
Analyte : Fe^{2+}, Cu^{2+}
Indicator : Ferroin

BROMATOMETRY

Titrant : Potassium Bromate
Analyte : Sb, As
Indicator : Methyl Red

IODIMETRY

Titrant : Iodine Solution
Analyte : Sodium thiosulphate
Indicator : Starch solution

DICHROMETRY

Titrant : Potassium dichromate
Analyte : Iron salts ($FeSO_4$)
Indicator : Sodium diphenylamine sulphate.

IODOMETRY

Titrant : Sodium thiosulphate
Analyte : Iodine (Prepared)
Indicator : Starch solution

TITRATION WITH POTASSIUM IODATE

Titrant : Potassium Iodate
Analyte : Vitamin-C, $CuSO_4$.
Indicator : Starch-Iodine solution.

Iodimetry & Iodometry

- Titration that involves iodine are reffered as iodine titration.
- Iodine acts as as a Mild or Weak Oxidizing Agent.
- Two types of iodine titrations are possible :

① Iodimetry
② Iodometry

Iodimetry

- Iodimetry is a type of Direct Titration.
- It is a type of redox titration.
- These are the titration in which free iodine is used.

- The titration in which standard iodine solution is used for the determination of reducing agents like Sodium Thiosulphate ($Na_2S_2O_3$) is known as Iodimetry.
- End Point of the titration is determined by change in colour from Blue to Colourless.

Methodology

- First iodine is mixed with kI because generally it is not easily soluble in water

$$kI + I_2 \longrightarrow kI_3$$

- Now we use starch solution as an indicator , iodine will reacts with starch and forms iodine - starch complex which appears blue in colour.

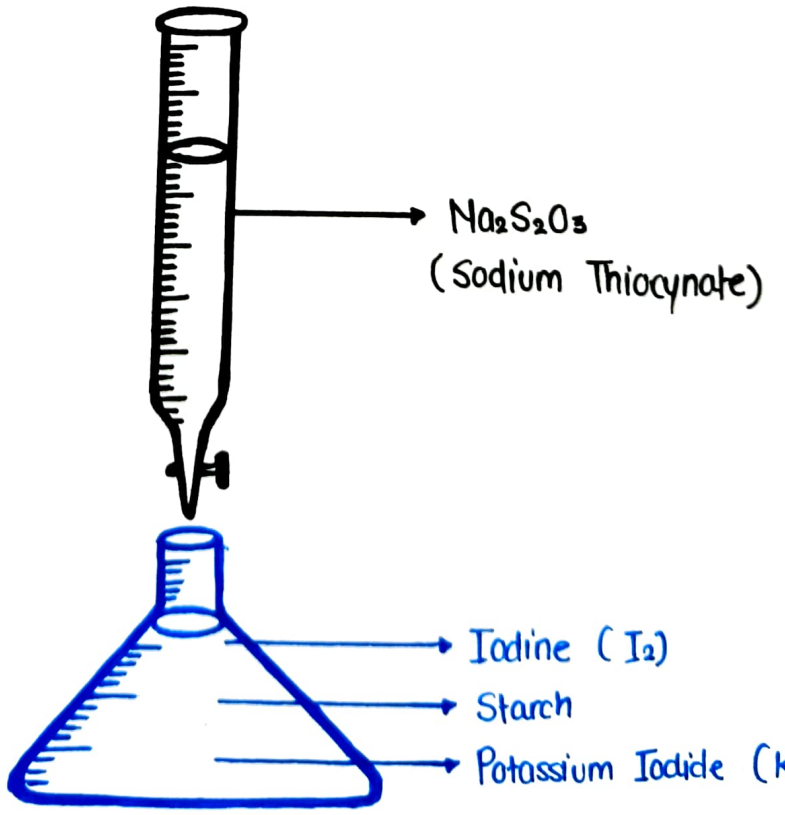
$$I_2 + \text{Starch} \longrightarrow \text{Blue Colour}$$

- Now this standard iodine solution is titrated with reducing agent ($Na_2S_2O_3$)

$$I_2 + 2S_2O_3^{2-} \rightleftharpoons S_4O_6^{2-} + 2I^-$$

- Now at the endpoint all iodine reacts with thiosulphate ions and converted into iodide and blue colour disappears.

$$I^- + \text{Starch indicator} \longrightarrow \text{No Colour}$$



IODOMETRY

- Iodometoy is a type of indirect titration.
- It is a type of redox titration
- These are the titration in which liberated iodine is used.

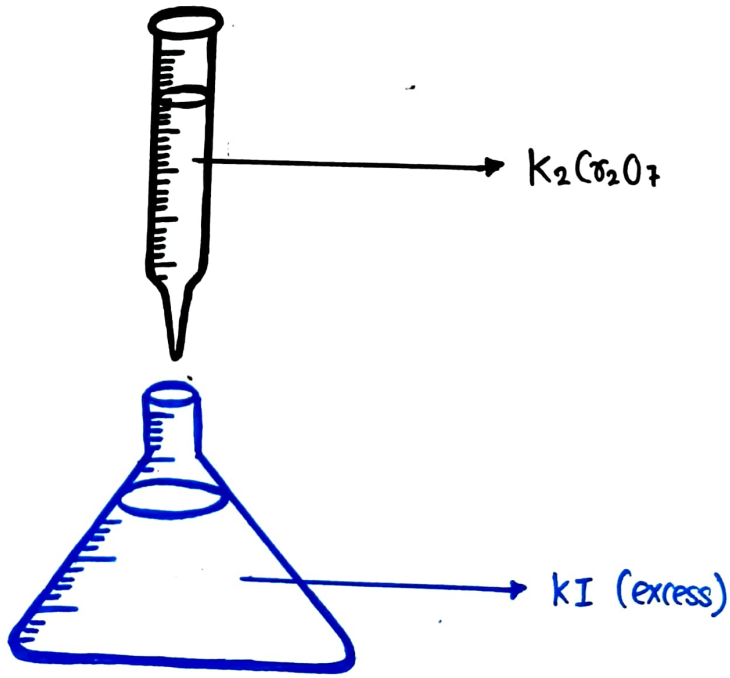
- When a standard solution of sodium thiocynate ($Na_2S_2O_3$) is used foo the deteomination of liberated iodine using starch solution as an indicatoo then it is known as Iodometoy.

- Iodometry is actually completed in two steps :

STEP - I

- The first step is done by the reaction between the oxidizing agent ($K_2Cr_2O_7$) and excess KI and as a result of the reaction iodine gets liberated.

$$K_2Cr_2O_7 + 6KI + 7H_2SO_4 \longrightarrow Cr_2(SO_4)_3 + 4K_2SO_4 + \mathbf{3I_2} + 7H_2O$$



STEP - II

- In step - II , ~~Sta~~ first starch solution is added in the liberated iodine which foom blue colour

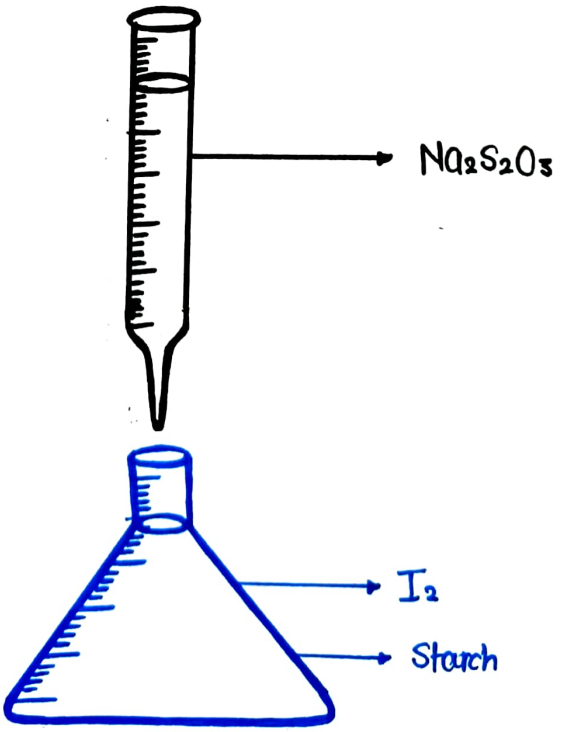
$$I_2 + \text{Starch} \longrightarrow \text{Blue Colour}$$

- Now we use standard sodium thiocynate solution which reacts with liberated iodine in the solution

$$I_2 + 2S_2O_3^{2-} \rightleftharpoons S_4O_6^{2-} + 2I^-$$

- Now at the end point all the liberated iodine reacts with thiosulphate and prooduce iodide ion , the indicator don't show any reaction with iodide ions , hence blue colour of solution disappears (colourless)

$$I^- + \text{Indicator} \longrightarrow \text{Colourless}$$



Major difference between Iodimetry and Iodometoy

IODIMETRY	IODOMETRY
• Iodine is taken directly in the analysis	• Iodine is prooduced as a result of Redox Reaction
• Direct Titration	• Indirect Titration
• Only 1 Redox Reaction	• Two Redox Reaction
• Iodine only gets reduced	• Iodine first get oxidized then get reduced
• Less Common Method	• More common method

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