Practical Pharmaceutical Analytical Chemistry - II

Second Level
First Semester 2018-2019

Section 2

Oxidation - Reduction Titration

(Redox Titration)

Experiment 1

Determination of Hydrogen Peroxide (H_2O_2)

(Official in USP and BP)

Principle

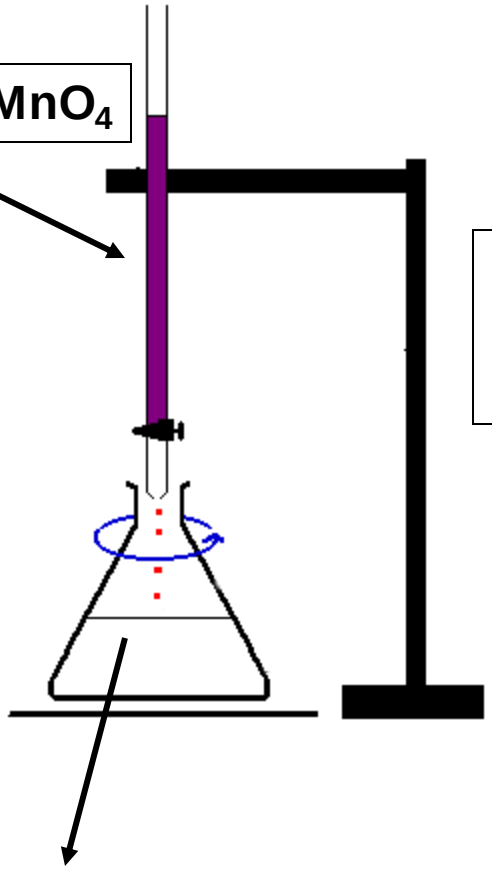
- ✓ By direct titration of the sample against standard $KMnO_4$ in presence of dil. H_2SO_4 .

$$5\ H_2O_2 + 2\ MnO_4^- + 16\ H^+ \longrightarrow 2\ Mn^{2+} + 8\ H_2O + 5\ O_2$$

- ✓ When potassium permanganate solution is added to hydrogen peroxide solution acidified with sulphuric acid, the permanganate is readily reduced into manganous salt while peroxide is oxidized into water and molecular oxygen.
- ✓ KMnO4 acts as a self-indicator for end point detection.

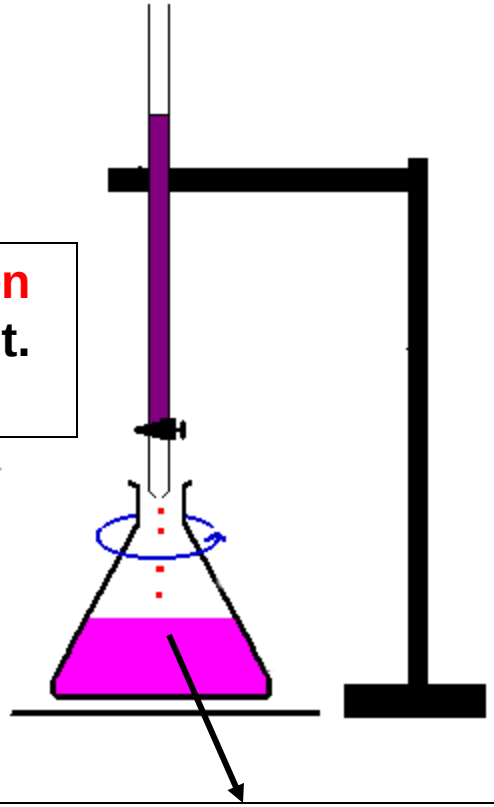
Procedure

0.1 N St. $KMnO_4$



10 ml Hydrogen peroxide + 5 ml dil. H_2SO_4

Titrate Hydrogen peroxide with St. KMnO4



At the E.P. the color in the conical flask changes from colorless to the first faint pink color persistent for about ½ min. with shaking

Calculations

Q. Calculate concentration of H_2O_2 in Normal, Molar and g % or % W / V ?

1. In Normal:

$$N \cdot V\ (KMnO_4) = N` \cdot V`\ (H_2O_2)$$

N = 0.1

V = E.P

N` = ??

V` = 10

2. In Molar:

$$N = M * n$$

$$M = N / n$$

Where n is the No. of electron transfer, n for H_2O_2 = 2

So, M = N / 2

3. In g %:

Conc. in g % = M * M.Wt / 10

Volume strength of H_2O_2

Hydrogen peroxide solution is a widely used antiseptic preparation. It is available in pharmacies in 3 concentrations:

10 Volume – **20** Volume – **30** Volume

≡ 3 g% – 6 g% – 9 g%

Volume strength of H_2O_2

It is the number of mLs of O_2 liberated by complete thermal decomposition of 1 mL of H_2O_2 solution at normal temperature and pressure (N.T.P) i.e: 25°C and 1 atm.

Example:

If concentration of H_2O_2 is 3%, what is its volume strength?

Step 1:

3 g H_2O_2 ⟶ 100 mL solution of H_2O_2

? g H_2O_2 ⟶ 1 mL solution of H_2O_2

Answer: 0.03 g H_2O_2 is present in 1 mL of 3% H_2O_2 solution

Step 2:

$2\ H_2O_2 \xrightarrow{\text{Thermal decomposition}} O_2 + 2\ H_2O$

2 moles H_2O_2 ⟶ 1 mole O_2

(2 x 34) g H_2O_2 ⟶ 22.4 liters O_2 at N.T.P

68 g H_2O_2 ⟶ 22400 mL O_2

0.03 g H_2O_2 ⟶ X mL O_2

X = **10 mL O_2**

Summary:

1 mL of 3% H_2O_2 solution (0.03 g H_2O_2)

by decomposition

10 mL O_2

3% H_2O_2 solution ≡ 10 volume H_2O_2 solution.

Experiment 2

Determination of Ferrocyanide ($K_4[Fe(CN)_6].3H_2O$)

Principle

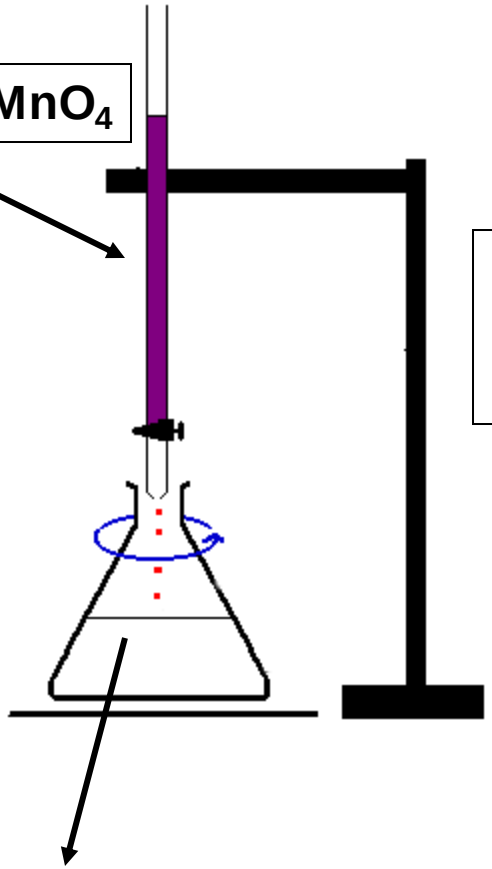
- ✓ By direct titration of the sample against standard $KMnO_4$ in presence of dil. H_2SO_4 .

$$5\,[Fe(CN)_6]^{4-} + MnO_4^- + 8\,H^+ \longrightarrow 5\,[Fe(CN)_6]^{3-} + Mn^{2+} + 4\,H_2O$$

- ✓ When potassium permanganate solution is added to Potassium Ferrocyanide solution acidified with sulphuric acid, the permanganate is readily reduced into manganous salt while Ferrocyanide is oxidized into Ferricyanide.
- ✓ $KMnO_4$ acts as a self-indicator for end point detection.

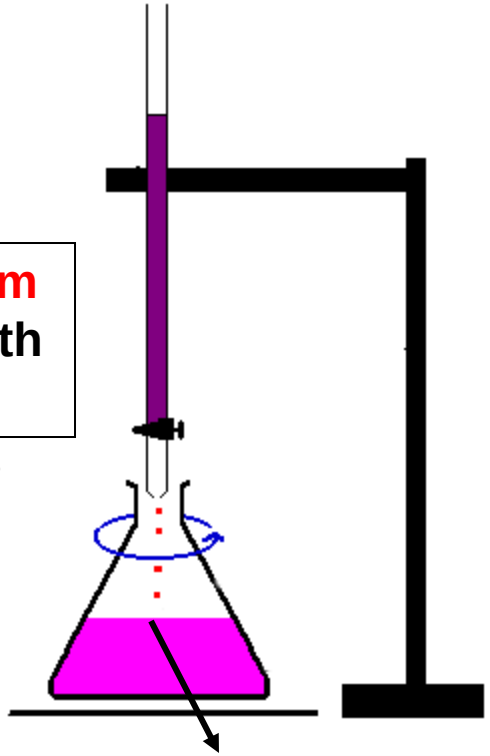
Procedure

0.1 N St. $KMnO_4$



10 ml Potassium Ferrocyanide + 5 ml dil. H_2SO_4

Titrate Potassium Ferrocyanide with St. KMnO4



At the E.P. the color in the conical flask changes from greenish yellow color to the first yellowish pink color persistent for about ½ min. with shaking

Calculations

N . V ($KMnO_4$) = N`. V` (Potassium Ferrocyanide)

N = 0.1 **N` = ??**

V = E.P **V` = 10**

Thank You