## **EXPERIMENT NO. 2**

**Aim:** To determine the amount of Oxalic acid and H<sub>2</sub>SO<sub>4</sub> present in 1 L. of solution **by** using N/10 NaOH and N/10 KMnO<sub>4</sub> solution.

Apparatus required: Burette, pipette, beakers, titration stand, conical flask and glass funnel etc.

Chemicals Required: NaOH, Oxalic acid, H<sub>2</sub>So<sub>4</sub>, Phenolphthalein and KMnO<sub>4</sub>.

**Theory:** This involves double titration.

1st Titration- Mixture (H<sub>2</sub>SO<sub>4+</sub>Oxalic Acid) Vs NaOH:

NaOH reacts with oxalic acid as well as H<sub>2</sub>SO<sub>4</sub> and find out the normality of oxalic acid and sulphuric acid. Reactions involves

- 1. (COOH)<sub>2</sub> 2 H<sub>2</sub>O + NaOH (COONa)<sub>2</sub> + 4 H<sub>2</sub>O
- 2. H<sub>2</sub>SO<sub>4</sub> + 2 NaOH Na<sub>2</sub>SO<sub>4</sub> + 2 H<sub>2</sub>O

By titrating NaOH against the mixture, we find out the total normality of mixture. 2nd Titration-Mixture ( $H_2SO_{4+}$  Oxalic Acid) Vs KMnO<sub>4</sub>: The mixture of solution is treated with KMnO<sub>4</sub> solution which will react with oxalic acid in the presence of  $H_2SO_4$ . By this titration we find out the normality and strength of oxalic acid.

$$60^{\circ} - 70^{\circ}$$
C

2. 
$$(COOH)_2 + [O] 2 CO_2 + H_2O] x 5$$

Complete reaction -

$$2 \text{ KMnO}_4 + 3H_2SO_4 + 5 (H_2C_2O_4) K_2SO_4 + 2MnSO_4 + 8H_2O + 10 CO_2$$

#### Procedure:

i)Titration of mixture (Oxalic Acid +H<sub>2</sub>SO<sub>4</sub>) and NaOH

- 1. Fill NaOH solution in the burette.
- 2. Now pipette out 10 ml of mixture (Oxalic Acid  $+H_2SO_4$ ) in the conical flask.
- 3. Add 2-3 drops of Phenolphthalein indicator in the conical flask.
- 4. Now start pouring NaOH in the flask till the colourless solution in the flask turns to pink. And note the volume of NaOH used.
- 5. Repeat the experiment for concordant readings.
- ii)Titration of Mixture (Oxalic Acid + H<sub>2</sub>SO<sub>4</sub>) and KMnO<sub>4</sub>:
  - 1. Fill 50 ml of KMnO₄ solution in the burette. Since it is coloured solution we note upper meniscus

for taking the initial reading.

- 2. Now pipette out 10 ml of mixture (Oxalic Acid +  $H_2SO_4$ ) in the conical flask and add 5 ml of  $H_2SO_4$  in the flask.
- 3. Then heat the conical flask up to  $60^{\circ}$   $70^{\circ}$ C.
- 4. Now start pouring KMnO<sub>4</sub> solution in the conical flask till the colourless solution in the flask turns to pink. And note the volume of KMnO4 used.
- 5. Repeat the experiment for concordant readings.

#### **Observations:**

### Titration i)

Solution in burette - NaOH

Solution in conical flask – (Oxalic Acid + H<sub>2</sub>SO<sub>4)</sub>

Indicator - Phenolphthalein

End Point – Colourless to Pink

Serial no.	Burette Reading		Volume Used(ml)
	Initial	Final	
1	0	28	28
2	0	29	29
3	0	29	29

# Titration ii)

Solution in burette – KMnO<sub>4</sub>

Solution in conical flask - (Oxalic Acid + H<sub>2</sub>SO<sub>4</sub>)

Indicator - KMnO<sub>4</sub>

End Point – Colourless to Pink

Serial no.	Burette Reading		Volume Used(ml)
	Initial	Final	
1	0	10.2	10.2
2	10.2	20.4	10.2
3	20.4	30.6	10.2

### **Calculations:**

### Titration- i)

#### **Mixture Vs NaOH**

 $N_1 \times V_1 = N_2 \times V_2$ (Mix.) (NaOH)  $N_1 = N_2 \times V_2 / V_1$   $= 0.1 \times 29 / 10$  $N_1 \text{(Mix.)} = 0.29 \text{ N}$ 

### Titration ii)

### Mixture Vs KMnO<sub>4</sub>

(Here, only Oxalic acid is titrated with KMnO<sub>4</sub>)  $N_1' \times V_1' = N_2' \times V_2'$ (Oxalic acid) (KmnO<sub>4</sub>)  $N_1' = N_2' \times V_2' / V_1'$   $N_1' = 0.1 \times 10.2 / 10$   $N_1'(Oxalic acid) = 0.102 \text{ N}$ Normality of  $H_2SO_4 = N_1(Mix.) - N_1'(Oxalic acid) = 0.29 - 0.102 \text{ N}$  = 0.188 NStrength of oxalic acid = N1' x Eq.wt.  $= 0.102 \times 63$  = 6.426 g/LStrength of  $H_2SO_4 = 0.188 \times Eq.wt$ .  $= 0.188 \times 49$ 

#### **Result:**

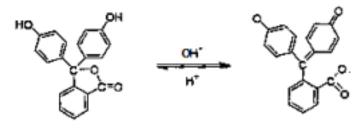
Oxalic Acid in the given mixture = 6.426g/L. H<sub>2</sub>SO<sub>4</sub> in the given mixture = 9.212 g/L

## **Structures:**

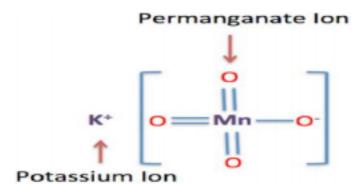
#### Phenolphthalein

= 9.212 g/L

## Colourless (Acidic Medium) Pink (Basic Medium)



#### Structure of KMnO<sub>4</sub>



### **Precautions:**

- 1. All the apparatus should be washed before use.
- 2. For measurement of coloured solution check the upper and lower meniscus of solution.
- **3.** Rinse the burette with  $KMnO_4$  solution and pipette with the given solution. **4.** Always use freshly prepared  $KMnO_4$  solution.

### **Applications:**

- 1. It is used to determine the amount of various nutrients present in a particular food item.
- 2. It is used in innovation of medical sciences.