## **Exp. 5:**

## **Titration of Oxalic Acid with KMnO4**

The titration of potassium permanganate (KMnO<sub>4</sub>) against oxalic acid (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>) is an example of redox titration. In close proximity to the endpoint, the action of the indicator is analogous to the other types of visual colour titrations in oxidation-reduction (redox) titrations.

#### Aim:

To determine the strength of potassium permanganate by titrating it against the standard solution of 0.1M oxalic acid.

### **Theory:**

Potassium permanganate is a strong oxidising agent and in the presence of sulfuric acid it acts as a powerful oxidising agent. In acidic medium the oxidising ability of KMnO<sub>4</sub> is represented by the following equation.

#### In acidic solution,

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

Solution containing  $MnO_4^-$  ions are purple in colour and the solution containing  $Mn^{2+}$  ions are colourless and hence permanganate solution is decolourised when added to a solution of a reducing agent. The moment there is an excess of potassium permanganate present the solution becomes purple.

Thus, KMnO<sub>4</sub> serves as self-indicator in acidic solution.

Potassium permanganate is standardized against pure oxalic acid. It involves a redox reaction. Oxalic acid is oxidised to carbon dioxide by KMnO<sub>4</sub>, which itself gets reduced to MnSO<sub>4</sub>. Oxalic acid reacts with potassium permanganate in the following way.

The chemical reaction at room temperature is given below.

Reduction Half reaction: 
$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

Oxidation Half reaction:- 
$$5(COOH)_2 + 5[O] \rightarrow 5H_2O + 10CO_2\uparrow$$

The *overall reaction* takes place in the process is.

Overall reaction:- 2KMnO<sub>4</sub> + 3H<sub>2</sub>SO<sub>4</sub> + 5(COOH)<sub>2</sub> 
$$\rightarrow$$
 K<sub>2</sub>SO<sub>4</sub> + 2MnSO<sub>4</sub> + 8H<sub>2</sub>O + 10CO<sub>2</sub> $\uparrow$ 

The *ionic equation* involved in the process is given below.

Reduction Half reaction:- [MnO<sub>4</sub>
$$^-$$
 + 8H $^+$  + 5e $^ \rightarrow$  Mn<sup>2+</sup> + 4H<sub>2</sub>O] x 2

Oxidation Half reaction:- 
$$[C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-] \times 5$$

Overall Ionic reaction: 
$$-2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$$

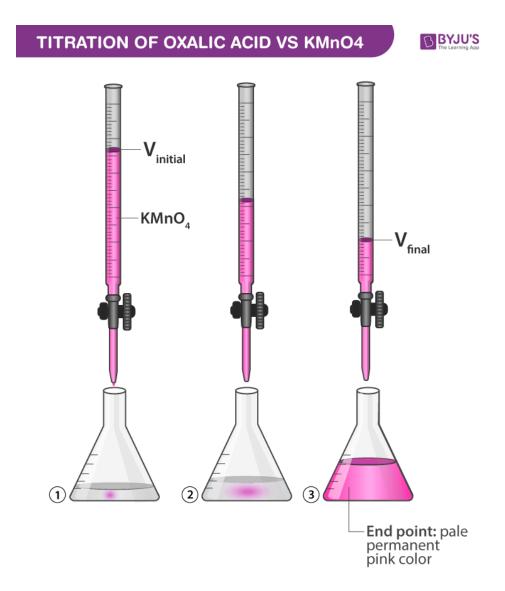
This titration cannot be carried out in the presence of acids like nitric acid or hydrochloric acid because itself is an oxidising agent. So hydrochloric acid chemically reacts with KMnO<sub>4</sub> solution forming chlorine which is also an oxidising agent.

## **Materials Required:**

- 1. Oxalic acid
- 2. Potassium permanganate solution
- 3. 1.0M sulphuric acid
- 4. Chemical balance
- 5. Burette
- 6. Burette stand.
- 7. Pipette
- 8. Conical flask
- 9. Funnel
- 10. Measuring flask
- 11. Weighing bottle
- 12. Magnetic stirrer with hot plate.

### **Apparatus Setup:**

- 1. In burette KMnO<sub>4</sub> solution
- 2. In Conical flask 10ml of oxalic acid + sulphuric acid
- 3. Indicator Self indicator (KMnO<sub>4</sub>)
- 4. End Point Appearance of permanent pale pink colour.



#### **Procedure:**

#### (a) Preparation of 0.1M standard solution of oxalic acid:

To determine the strength of potassium permanganate by titrating it against the standard solution of 0.1M oxalic acid." we should calculate for 0.1 M of oxalic acid.

#### The molecular mass of oxalic acid = 126 g

For the preparation of 100 ml of M/10 oxalic acid solution amount of oxalic acid required = 1.57 g

- 1. Weigh an empty watch glass using a chemical balance.
- 2. Weigh 1.57g of oxalic acid accurately in the watch glass.
- 3. With the help of a funnel transfer the oxalic acid into the measuring flask.
- 4. Now wash the funnel with distilled water without removing the funnel from the flask.
- 5. Make the solution up to the marked point in 100 ml volumetric flask with distilled water and make sure the oxalic acid is fully dissolved.

# (b) Preparation of 0.1M standard solution of potassium permanganate KMno4:

1. 3.16 g of potassium permanganate KMnO4 should be dissolved in 100 ml in volumetric flask.

Fill up the prepared KMnO4 in the burette.

# (c) Titration of potassium permanganate solution against standard oxalic acid solution:

- 1. Rinse the burette with the potassium permanganate solution and fill the burette with potassium permanganate solution.
- 2. Fix the burette in the burette stand and place the white tile below the burette in order to find the end point correctly.
- 3. Pipette out diluted sulphuric acid (20 ml) to 20 ml (Oxalic acid solution) in conical flask in order to prevent oxidation of manganese to form manganese dioxide.
- 4. Heat the mixture up to 60-70°C before titrating with potassium permanganate.
- 5. Note down the initial reading in the burette before starting the titration.
- 6. The hot solution is titrated against potassium permanganate solution and simultaneously swirl the solution in the flask gently.
- 7. Initially the purple colour of KMnO<sub>4</sub> is discharged with oxalic acid. The appearance of permanent pink colour reveals the end point.
- 8. Note down the upper meniscus on the burette readings. Record the reading in the observation table given below in order to calculate the molarity of KMnO<sub>4</sub> given.

#### **Calculations:**

To calculate the strength of given KMnO<sub>4</sub> in terms of molarity the following formula is used.

$$a_1M_1V_1 = a_2M_2V_2$$

Where a<sub>1</sub> and a<sub>2</sub> are stoichiometric coefficient of oxalic acid and KMnO<sub>4</sub> in a balanced chemical equation.

$$a_1 = 2$$

$$a_2 = 5$$

Where  $M_2$  and  $M_1$  are molarities of potassium permanganate and oxalic acid solutions used in the titration

 $V_2$  and  $V_1$  are the volume of potassium permanganate and oxalic acid solutions used in the titration.

#### Therefore,

 $KMnO_4 = Oxalic acid$ 

$$5M_2V_2 = 2M_1V_1$$

$$M_2 = (2M_1V_1/5M_2V_2)$$

The strength of KMnO<sub>4</sub> is calculated by using the molarity.

Strength = Molarity x Molar mass

#### **Results and Discussion:**

- 1. Molarity of KMnO<sub>4</sub> is \_\_\_\_\_
- 2. The Strength of KMnO<sub>4</sub> is \_\_\_\_\_M.

## **Questions**

- In this titration of KMnO4 vs oxalic acid, what is the indicator used?
- What is meant by endpoint?
- What is meant by redox titration?
- Why we add diluted sulfuric acid to oxalic acid in Redox titration.