Exp No: 07

Name of the Experiment: Standardization of potassium permanganate ($KMnO_4$) Solution with standard oxalic acid ($C_2H_2O_4.2H_2O$) Solution [REDOX Titration]

1. Theory:

Primary standard substance is a highly pure (~99%) and chemically stable substance. Dissolving in a known volume of solvent, primary standard substance prepares a precise concentration solution known as primary standard solution. It has significant application as a reference in chemical analysis and instruments calibration. For examples, the use of primary standard solution in titrations and other analytical techniques to determine the concentration of an unknown substance. Unlike primary standards, secondary standard substance may have a slightly lower degree of purity and stability, as they are often hygroscopic or highly reactive towards air oxygen. To ensure the accuracy of analytical measurements, the secondary standard reagents needed to be standardized or calibrated before its use in any analysis method. The technique that involves the accurate determination of the concentration of the secondary standard reagent by reacting it with another substance of known concentration (termed as titrant) is referred as standardization.

In this experiment, a secondary standard reagent, potassium permanganate solution will be standardized by a standard oxalic acid solution, which is a primary standard substance. The standardization of potassium permanganate using oxalic acid involves a redox reaction where the analyte ($KMnO_4$) function as oxidizing agent and the titrant (oxalic acid) as a reducing agent, resulting in change in corresponding oxidation states. Like, in acidic medium, $KMnO_4$ accepts 5 electrons and is reduced to Mn^{+2} and H_2O.

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

The molecular reaction of oxalic acid and potassium permanganate is as follows:

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

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

Overall reaction: $2KMnO_4 + 5\ H_2C_2O_4 + 8\ H_2SO_4 \rightarrow \mathbf{K_2SO_4} + 2\ MnSO_4 + 5\ H_2SO_4 + 10\ CO_2 + 8\ H_2O$

The potassium permanganate solution is purple in colour owing to the presence of MnO_4^- ion. This purple colour may disappear with the reduction MnO_4^- ion to Mn^{+2} ion. Thus, $KMnO_4$ can act as a self-indicator in acidic solution, where the change of solution color may indicate the end point or completion of the redox reaction. Therefore, the concentration of the analyte ($KMnO_4$) is calculated based on the stoichiometry of the balanced redox equation and the volume and concentration of the titrant used. From the stoichiometry of the above redox reaction we can write

$$2\ \text{mol}\ KMnO_4 \equiv 5\ \mathbf{mol}\ \mathbf{H_2C_2O_4}$$

As the kinetics of reduction of $KMnO_4$ is comparatively slow, often heat is applied to catalyze the reaction at initial phase

2. Apparatus:

Volumetric flask (100mL), Conical flask (250mL), Burette, Pipette (10mL), Funnel, Wash Bottle, Burner, Iron Stand

2. Chemicals:

1. Oxalic Acid Dihydrate ($C_2H_2O_4.2H_2O$)
2. Potassium Permanganate ($KMnO_4$)
3. Sulfuric Acid (H_2SO_4)

4. Phase:

The experiment will be done in two phases

a. Preparation of standard 0.1N $H_2C_2O_4.2H_2O$ (oxalic acid) solution
b. Standardization of unknown concentration potassium permanganate ($KMnO_4$) solution

5. Procedure:

a. Preparation of standard 0.1 N $C_2H_2O_4.2H_2O$ Solution

To prepare 0.1 N 100 mL $C_2H_2O_4.2H_2O$ solution, weigh out the required amount (calculate by yourself) of $C_2H_2O_4.2H_2O$ and transfer into a 100 mL volumetric flask, dissolve it properly and make up to the mark by adding distilled water.

$$\text{Strength of } C_2H_2O_4 \text{ Solution} = \frac{wt.taken}{wt.\ to\ be\ taken} \times 0.1 \text{ N}$$

$$= \qquad \text{N}$$

b. Standardization of approx potassium permanganate ($KMnO_4$) with standard oxalic acid ($C_2H_2O_4.2H_2O$)

1. Rinse and fill the burette with potassium permanganate ($KMnO_4$) solution
2. Pipette out 10 mL di-hydrate oxalic acid ($C_2H_2O_4.2H_2O$) solution in a conical flask
3. Dilute the solution up to 100 ml with distilled water.
4. Add 10 mL (3.0 N) of sulphuric acid (H_2SO_4) in the solution.
5. Heat the mixture upto 60°C temperature before titrating with potassium permanganate (Check the temperature using a thermometer).
6. Slowly titrate the oxalic acid solution with potassium permanganate ($KMnO_4$) solution and simultaneously gently swirl the solution in the flask.

7. The purple colour of $KMnO_4$ disappears when it added to oxalic acid solution The appearance of permanent pink colour will indicate the end point of titration, i.e., the complete oxidation of oxalic acid

6. Experimental Data:

Table for the titration of $KMnO_4$

Number of Observation	Volume of $C_2H_2O_4$ solution, V_1 (mL)	Volume of $KMnO_4$ solution				Remarks
		IBR (mL)	FBR (mL)	Diff. (mL)	Mean Volume, V_2 (mL)	
1.						
2.						
3.						
4.						

N.B.: IBR = Initial Burette Reading, FBR = Final Burette Reading

7. Calculation:

$$S_2 = V_1S_1/V_2$$

= ---------- N solution

= N

Where,

V_1 (Volume of $C_2H_2O_4$ solution) = mL

S_1 (Strength of $C_2H_2O_4$ solution) = N

V_2 (Volume of $KMnO_4$ solution) = mL

S_2 (Strength of $KMnO_4$ solution) = ?

8. Result:

The Strength of $KMnO_4$ solution is ----------N

9. % of error:

[(Std result ~ Obs result) / Std result] × 100

= %

10. Discussion

[Write down your ***Observation, Assumption, Perception, Limitations, Safety Considerations, and Possible source of Error*** regarding the experiment]

11. Key Points:

a. Electronic concept of Oxidation and Reduction reaction

b. Define: Oxidizing agent and Reducing agent

c. Write down the Arrhenius Equation that shows the dependency of reaction rate on the temperature

d. Define activation energy. Draw activation energy diagram for Exothermic and Endothermic Reactions

e. Define catalyst of a reaction. Discuss the mechanism of catalyst on a reaction using activation energy diagram

f. Define auto catalyst and self-indicator

g. Why do we use H_2SO_4 for this titration instead of HCl or HNO_3?