



FACULTY OF
**HUMANITIES
& SCIENCES**

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Module Code	SC1162
Title of Experiment	Standardization of potassium permanganate using Na ₂ C ₂ O ₄ solution
Number of the Experiment	05
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BSc (Hons) Biotechnology

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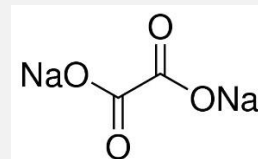
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Objective:- To observe a reduction oxidation process
To gain experience with stoichiometry in balanced reaction
To understand quantitative analysis
To use primary standard solutions to determine the concentration of potassium permanganate solutions in titrimetric.

Introduction:-

Titration is an analytical method used to determine the exact amount of a substance by reacting that substance with a known amount of unknown amount. The completed reaction of a titration is usually indicated by a color change of end point.

The procedure generally used in the standardization of permanganate solutions with sodium oxalate is that called oxidation reduction titration or redox titration. Potassium permanganate's formula is KMnO_4 . Its color is deep purple color and it is soluble in water. Sodium oxalate is the sodium salt of oxalic acid with the formula $\text{Na}_2\text{C}_2\text{O}_4$. It is a white crystalline solid. Sodium oxalate may be used as a primary standard for standardizing potassium permanganate solution.



In the case of potassium permanganate and sodium oxalate, potassium permanganate is reduced while sodium oxalate is oxidized. More specifically, carbon from the oxalate anion loses electrons becoming oxidized while the manganese atom gains electrons and becomes reduced. In this external indicator is not used because potassium permanganate (KMnO_4) is a popular titrant because it serves as its self indicator in acidic solution. Sulfuric acid or H_2SO_4 is used in this experiment because to form acidic medium and it is stable towards oxidation.

It dissolves in water as a strong electrolyte to give K^+ and MnO_4^- ions. Manganese is in the +7 oxidation the highest state in KMnO_4 . Therefore, it can only be reduced to lower oxidation states in redox reactions. Although it is possible to make +4, +3, 0 and other oxidation states, the most common reaction is a five-electron reduction to +2; that is Mn^{2+} which occurs as a hydrated ion in water and it is light pink color. The reduction half reaction is,



$\text{Na}_2\text{C}_2\text{O}_4$ has weak base properties, and in strong acid it is immediately converted to oxalic acid and carbon is in the +6 oxidation state. The sodium form dissolves to give 2 Na^+ and one oxalate ($\text{C}_2\text{O}_4^{2-}$) a trio of ions. The oxidation half reaction may then be written as,



The two half reactions have different numbers of electrons, five for the reduction and two for the oxidation. The rules for balancing redox reactions include that electron must be canceled out when adding and stoichiometry are done. If the first reaction is multiplied by 2 and the second by 5, then the two are added together we get the balanced, overall redox reaction,



This reaction tells us that five oxalate ($\text{C}_2\text{O}_4^{2-}$) and two permanganates (MnO_4^-) on a mole basis go together to make products in the presence of a lot of acid. When that occurs, the following equation must be true for the above overall redox reaction:

2 mol permanganate = 5 mol oxalate

This equation tells us that if you want to convert moles of permanganate into moles of oxalate (stoichiometry) you must multiply by 5/2.

Material:- Oxalic acid solution	Spatula	Glass rod	Burette
KMnO ₄ solution	Beaker	Funnel	Burette stand
Dil. H ₂ SO ₄	Pipette	Test tube	Hot plate
Distilled water	Titration flask	Dropper	

Method/ procedure:-

Preparation of 5.0×10^{-2} mol dm⁻³ Na₂C₂O₄ solution

0.67g mass of Na₂C₂O₄ was calculated to prepare a volume of 100.00 cm³ of 5.0×10^{-2} mol dm⁻³ Na₂C₂O₄ solution. The calculated mass of Na₂C₂O₄ was transferred to a 100.00 cm³ volumetric flask. A small volume of distilled water was added and swirled to dissolve the solid completely. Using distilled water was diluted up to the mark.

Standardization of potassium permanganate.

Firstly the burette was rinsed with tap water and with distilled water. Next the burette was rinsed with KMnO₄ solution. The burette was filled with 2.0×10^{-2} mol dm⁻³ KMnO₄ solution and the initial burette reading was recorded. A volume of 10.00 cm³ of Na₂C₂O₄ solution was transferred to a titration flask by using pipette. 5 cm³ of dilute H₂SO₄ was added. The solution to 60 °C was heated and titrated with the KMnO₄ solution until the end point was reached. End point is usually defined as the first persistent pink color which lasts for about 30 seconds. The same procedure was repeated using 10.00 cm³ of distilled water instead of Na₂C₂O₄ solution. With KMnO₄ solution was titrated until the end point was reached. Then the experiment was repeated three times.

Result:-**Titration between KMnO₄ and Na₂C₂O₄**

Concentration of Na₂C₂O₄ = $5.0 \times 10^{-2} \text{ mol dm}^{-3}$

Volume of Na₂C₂O₄ solution = 10.00 cm³

Actual Concentration of KMnO₄ = $2.0 \times 10^{-2} \text{ mol dm}^{-3}$



Trial Number	Initial burette reading (cm ³) ±0.05 cm ³	Final Burette reading (cm ³) ±0.05 cm ³	Volume transferred (cm ³) ±0.05 cm ³
1	0.00	11.10	11.10
2	0.00	11.20	11.20
3	0.00	11.15	11.15

Blank Titration Readings

Trial Number	Initial burette reading (cm ³) ±0.05 cm ³	Final Burette reading (cm ³) ±0.05 cm ³	Volume transferred (cm ³) ±0.05 cm ³
1	0.00	0.05	0.05
2	0.00	0.05	0.05

Calculation:-

Calculation mass of sodium oxalate,

Na₂C₂O₄ concentration = Na₂C₂O₄ molar / Na₂C₂O₄ volume

$$5.0 \times 10^{-2} \text{ mol dm}^{-3} = \text{Na}_2\text{C}_2\text{O}_4 \text{ molar} / 100.00 \times 10^{-3} \text{ dm}^{-3}$$

$$\text{Na}_2\text{C}_2\text{O}_4 \text{ molar} = 5.0 \times 10^{-2} \times 100.00 \times 10^{-3}$$

$$= 5.0 \times 10^{-3} \text{ mol}$$

$$\text{Na}_2\text{C}_2\text{O}_4 \text{ mass} = \text{Na}_2\text{C}_2\text{O}_4 \text{ molar} \times \text{Na}_2\text{C}_2\text{O}_4 \text{ molar mass}$$

$$= 5.0 \times 10^{-3} \text{ mol} \times 134 \text{ g mol}^{-1}$$

$$= 0.67 \text{ g}$$

Concentration of NaOH solution can be calculated by using the equation.



$$5 : 2$$

Molar ratio of $\text{Na}_2\text{C}_2\text{O}_4$ and $\text{KMnO}_4 = 5 : 2$,

Concentration = molar (C) / volume (V)

$$\frac{\text{Na}_2\text{C}_2\text{O}_4 \text{ molar}}{\text{KMnO}_4 \text{ molar}} = \frac{5}{2} = \frac{\text{Na}_2\text{C}_2\text{O}_4 \text{ concentration (C1)} \times \text{Na}_2\text{C}_2\text{O}_4 \text{ volume (V1)}}{\text{KMnO}_4 \text{ concentration (C2)} \times \text{KMnO}_4 \text{ volume (V2)}}$$

Where, C1 and V1 are the concentration and volume of the $\text{Na}_2\text{C}_2\text{O}_4$ and C2 and V2 are the concentration and volume of the KMnO_4 .

$$\text{KMnO}_4 \text{ molar} = \text{Na}_2\text{C}_2\text{O}_4 \text{ molar}$$

$$\left. \begin{array}{l} 5 \times \text{KMnO}_4 \text{ concentration (C2)} \times \\ \text{KMnO}_4 \text{ volume (V2)} \end{array} \right\} = \frac{2 \times \text{Na}_2\text{C}_2\text{O}_4 \text{ concentration (C1)} \times}{\text{Na}_2\text{C}_2\text{O}_4 \text{ volume (V1)}}$$

Due to $\text{Na}_2\text{C}_2\text{O}_4$ concentration is $5.0 \times 10^{-2} \text{ mol dm}^3$ and $\text{Na}_2\text{C}_2\text{O}_4$ volume is 10.00 cm^3 ,

2 trial,

$$\begin{aligned} \text{KMnO}_4 \text{ volume} &= 11.20 \text{ cm}^3 - 0.05 \text{ cm}^3 \\ &= 11.15 \text{ cm}^3 \end{aligned}$$

Moles = Concentration (C) * Volume (V)

Due to molar ratio of $\text{Na}_2\text{C}_2\text{O}_4$ and $\text{KMnO}_4 = 5 : 2$

$$\left. \begin{array}{l} 5 \times \text{KMnO}_4 \text{ concentration (C2)} \times \\ \text{KMnO}_4 \text{ volume (V2)} \end{array} \right\} = \frac{2 \times \text{Na}_2\text{C}_2\text{O}_4 \text{ concentration (C1)} \times}{\text{Na}_2\text{C}_2\text{O}_4 \text{ volume (V1)}}$$

$$5 (C2 \times 11.15 \times 10^{-3} \text{ dm}^3) = 2 (5.0 \times 10^{-2} \text{ mol dm}^3 \times 10.00 \times 10^{-3} \text{ dm}^3)$$

$$\text{Concentration of KMnO}_4 = \frac{5.0 \times 10^{-2} \text{ mol dm}^3 \times 10.00 \times 10^{-3} \text{ dm}^3}{11.15 \times 10^{-3} \text{ dm}^3} \times \frac{2}{5}$$

$$= 1.794 \times 10^{-2} \text{ mol dm}^{-3}$$

$$= 1.8 \times 10^{-2} \text{ mol dm}^{-3}$$

3 trial,

$$\begin{aligned}\text{KMnO}_4 \text{ volume} &= 11.15\text{cm}^3 - 0.05\text{cm}^3 \\ &= 11.10 \text{ cm}^3\end{aligned}$$

Moles = Concentration (C) * Volume (V)

Due to molar ratio of $\text{Na}_2\text{C}_2\text{O}_4$ and $\text{KMnO}_4 = 5 : 2$

$$\frac{5 \times \text{KMnO}_4 \text{ concentration } (C_2) \times \text{KMnO}_4 \text{ volume } (V_2)}{2 \times \text{Na}_2\text{C}_2\text{O}_4 \text{ concentration } (C_1) \times \text{Na}_2\text{C}_2\text{O}_4 \text{ volume } (V_1)}$$

$$5 (C_2 * 11.10 * 10^{-3} \text{ dm}^3) = 2 (5.0 * 10^{-2} \text{ mol dm}^3 * 10.00 * 10^{-3} \text{ dm}^3)$$

$$\begin{aligned}\text{Concentration of KMnO}_4 &= \frac{5.0 * 10^{-2} \text{ mol dm}^3 * 10.00 * 10^{-3} \text{ dm}^3}{11.10 * 10^{-3} \text{ dm}^3} \times \frac{2}{5} \\ &= 1.801 * 10^{-2} \text{ mol dm}^{-3} \\ &= 1.8 * 10^{-2} \text{ mol dm}^{-3}\end{aligned}$$

In here, 1 trial's volume transferred and concentration can ignore. so, the second and third trials can be considered as final.

The average of the calculation is found by adding one and two and they dividing by two.

Average of concentration of $\text{KMnO}_4 = C_2 \text{ trial 2} + C_3 \text{ trial 3}$

$$\begin{aligned}&= \frac{(1.794 * 10^{-2} \text{ mol dm}^{-3} + 1.801 * 10^{-2} \text{ mol dm}^{-3})}{2} \\ &= 1.797 * 10^{-2} \text{ mol dm}^{-3} \\ &= 1.8 * 10^{-2} \text{ mol dm}^{-3}\end{aligned}$$

Difference between the experimental and actual concentration of KMnO_4 ,

= Actual concentration – Average concentration

$$= 2.0 * 10^{-2} \text{ mol dm}^{-3} - 1.8 * 10^{-2} \text{ mol dm}^{-3}$$

$$= 0.2 * 10^{-2} \text{ mol dm}^{-3}$$

$$= 2.0 * 10^{-3} \text{ mol dm}^{-3}$$

Standard deviation of the result,

$$\begin{aligned}
S_x &= \left(\left(\sum_{i=1}^n (x_i - \bar{x})^2 \right) / (n-1) \right)^{1/2} \\
&= \left[\frac{(2 \text{ trial con.} - \text{average con.})^2 + (3 \text{ trial con.} - \text{average con.})^2}{2-1} \right]^{1/2} \\
&= \sqrt{\frac{(1.794 \times 10^{-2} \text{ mol dm}^{-3} - 1.797 \times 10^{-2} \text{ mol dm}^{-3})^2 + (1.801 \times 10^{-2} \text{ mol dm}^{-3} - 1.797 \times 10^{-2} \text{ mol dm}^{-3})^2}{2-1}} \\
&= \left[25 \times 10^{-10} \text{ mol dm}^{-3} \right]^{1/2} \\
&= 5 \times 10^{-5} \text{ mol dm}^{-3}
\end{aligned}$$

Error the average of concentration,

$$\begin{aligned}
\% \text{ Error} &= \left[\frac{\text{theoretical value} - \text{experimental value}}{\text{Theoretical value}} \right] * 100\% \\
\% \text{ Error} &= \left[\frac{2.0 \times 10^{-2} \text{ mol dm}^{-3} - 1.8 \times 10^{-2} \text{ mol dm}^{-3}}{2.0 \times 10^{-2} \text{ mol dm}^{-3}} \right] * 100\% \\
&= 10\%
\end{aligned}$$

Discussion:-

Titration is important in chemistry as it allows for an accurate determination of solution concentration of the analyte.

During the course of the titration, the titrate KMnO_4 was added slowly to the $\text{Na}_2\text{C}_2\text{O}_4$ solution. If we added quickly we cannot accurately end point.

In here three trials were done to success the experiment. But in 1 trial, volume transferred is very different 2 trial. In 1 trial, KMnO_4 has been expended. so here, if there is only $\pm 0.05 \text{ cm}^3$ difference between the results of each trials, they included in the calculation to find concentration. So, 1 trial result was not added to final calculation.

When compare the actual value and experimental value, difference was very low. So due to that value is very small, the concentration of the experiment is close to the actual value. So, accuracy is acceptable in this experiment.

The standard deviation is directly related to precision of a procedure where low standard deviations are indicative of a high level of precision. So, in this experiment, standard deviation was very low. Due to that value was very small value and close to zero, precision of data is also high, and this experiment is precise. So, this experiment is successful.

KMnO₄ is not considered as a primary standard because it is difficult to obtain the pure state of KMnO₄ as it is not free from MnO₂. In this experiment, KMnO₄ is acted as its self-indicator. Due to Na₂C₂O₄ is initially colorless when as soon as the endpoint is reached and KMnO₄ is in excess, the solution has a permanent pink color. So, an external indicator was not necessary for this titration.

Na₂C₂O₄ solution was heated to 60 °C before starting the titration because without heating it is a slow process as an energy greater than activation energy is required for a reaction. When it was heated 60 °C, it was started sweat. Oxalate should not be heated to higher temperatures because overheating cause decomposition of oxalic acid as CO₂. The preheating up 60 °C during the titrate will provide the enough kinetic energy and activation energy and in here Mn⁺² acts as auto catalyst. It is enough to complete the experimental. So, it is not necessary to maintain the temperature at 60 °C throughout the titration.

Some of drops dilute sulfuric acid (H₂SO₄) was added to the titration flask. Titrations with Permanganate must be carried out in strong acid solution. Sulfuric Acid is generally used for this because it is stable towards oxidation. Hydrochloric acid (HCl) or Nitric acid (HNO₃) cannot be used in this because there can be reduce.

In here, a blank titration was did because to know exactly the volume of KMnO₄ consumed at the end point. The permanganate end point is not permanent because excess permanganate ion slowly reacts with the relatively large concentration of manganese (II) ions that are present at the end point. So blank titration volume should be deducted from trial volume.

In here adequate H₂SO₄ was added to the titration flask because if not KMnO₄ is not reduction into Mn²⁺. Then brown color precipitate of MnO₂ can be formed. In here when KHP was added from beaker to the volumetric flask so that it escapes along the glass rod. The limitation of the equipment was realized. Sufficient quantities were used of analytical and titration. The precision of the glassware was verified.

Conclusion:-

Thus, KMnO_4 serves as self-indicator in acidic solution. Potassium permanganate is standardized against pure oxalic acid. It involves a redox reaction. Oxalic acid is oxidized to carbon dioxide by KMnO_4 , which itself gets reduced to MnSO_4 .

Reference:-

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