Experiment Number: 07
Name of the experiment:  
_Standardization of Potassium Permanganate solution with Standard Sodium Oxalate Solution._

Course: Chem-114

Name: — Noor Nashid Islam
Roll: — 0105044
Group: — A2
Partner's Roll:—0105053
Department: — C.S.E.

Date of Performance: — 14-08-2002
Date of Submission: — 21-08-2002
Objective:—

The objective of this experiment is to determine the strength of Potassium Permanganate with a standard Sodium Oxalate solution. This reaction helps to study about oxidation and reduction theory. As, the color of potassium permanganate changes with the reaction, no further indicator is used in this experiment to determine the end point.

Theory:—

In this experiment, potassium permanganate solution is standardized with the help of standard sodium oxalate solution. The reaction that occurs here is oxidation and reduction reaction. An oxidation-reduction reaction (red ox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom). Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom).

Reaction involved in this reaction is:

\[ \text{MnO}_4^- + 8 \text{H}^+ + 5 e^- \rightarrow \text{Mn}^{2+} + 4 \text{H}_2\text{O} \]
\[ 2 \text{KMnO}_4 + 5 \text{Na}_2\text{C}_2\text{O}_4 + 8 \text{H}_2\text{SO}_4 \rightarrow \text{K}_2\text{SO}_4 + 2 \text{MnSO}_4 \]
\[ + 5 \text{Na}_2\text{SO}_4 + 10 \text{CO}_2 + 8 \]
In this reaction, $\text{MnO}_4^-$ is reduced to $\text{Mn}^{+2}$ and $\text{Na}_2\text{C}_2\text{O}_4$ is oxidized to $\text{CO}_2$.

The following equation is used to calculate the strength of Potassium Permanganate:

$$V_A \times S_A = V_B \times S_B$$

Here,

- $V_A$ = Volume of Potassium Permanganate
- $S_A$ = Strength of Potassium Permanganate
- $V_B$ = Volume of Sodium Oxalate
- $S_B$ = Strength of Sodium Oxalate

**INDICATOR AND WHY USED:**

The direct reaction is slow as one can see in a titration. The first few drops of permanganate added to the acidified oxalate solution are not decolorized immediately. $\text{Mn}^{+2}$ ions produced in the reaction acts as a catalyst. They react with permanganate to form intermediate oxidation states of manganese. These states, in turn, react rapidly with oxalate to give the products. So $\text{KMnO}_4$ acts as an auto catalyst in this reaction. This is the advantage of $\text{KMnO}_4$ is that it serves as its own indicator, the pink colour being distinguishable even if the solution is very dilute. Therefore no indicator is
used in this reaction.

**Apparatus:**

1. Conical flask
2. Burette
3. Pipette
4. Volumetric flask
5. Stand
6. Funnel

**Name of the chemicals used:**

1. Sodium Oxalate Solution \((Na_2C_2O_4)\)
2. \(H_2SO_4\) solution (2N)
3. Potassium Permanganate Solution \((KMnO_4)\)
4. Distilled water

**Data:**

**TABLE: 1**

(Standardization of Potassium Permanganate solution with Standard Sodium Oxalate solution)

<table>
<thead>
<tr>
<th>Burette</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial Reading</td>
<td>Final Reading</td>
<td></td>
</tr>
<tr>
<td>---</td>
<td>----------------</td>
<td>---------------</td>
<td></td>
</tr>
<tr>
<td></td>
<td>ml</td>
<td>ml</td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>1.0</td>
<td>11.7</td>
<td>10.7</td>
</tr>
<tr>
<td>02</td>
<td>11.7</td>
<td>22.4</td>
<td>10.7</td>
</tr>
<tr>
<td>03 (Not taken)</td>
<td>22.4</td>
<td>33.3</td>
<td>10.9</td>
</tr>
<tr>
<td>04</td>
<td>33.3</td>
<td>44.0</td>
<td>10.7</td>
</tr>
</tbody>
</table>

**Calculation:**

In this case weight taken = 0.739
Weight to be taken = 0.6701

So the strength of \( Na_2C_2O_4 \) = \((0.739 \times 0.1) / 0.6701 \) N
=0.11 N

From \( V_{\text{red}} \times S_{\text{red}} = V_{\text{ox}} \times S_{\text{ox}} \)

Here,

\[
\begin{align*}
V_{\text{Na}_2\text{C}_2\text{O}_4} &= 10 \text{ ml} \\
S_{\text{Na}_2\text{C}_2\text{O}_4} &= 0.11 \text{ N} \\
V_{\text{KMnO}_4} &= 10.7 \text{ ml} \\
S_{\text{KMnO}_4} &= ?
\end{align*}
\]

So,

\[
10 \times 0.11 = 10.7 \times S_{\text{KMnO}_4}
\]

\[
\Rightarrow \quad S_{\text{KMnO}_4} = 0.103 \text{ N}
\]

Result:

The Determined Strength of Potassium Permanganate Solution is:

\[
S_{\text{KMnO}_4} = 0.103 \text{ N}
\]

Percentage of error:
(Known Value - Observed Value) \times 100
\[
\text{Percentage of error} = \frac{\text{Known value}}{\text{Known value}}
\]

Known value = 0.105 N
Observed amount of iron = 0.103 N
Percentage of error = 1.9%

Precautions:

At first the burette was cleaned with distilled water carefully; then it was rinsed with rinse solution to avoid the density change of the given solution.

In the same way the pipette was also washed with distilled water and rinsed with the rinse solution.

All the apparatus were handled carefully and according to the rules.

The pipette was kept on a clean sheet of paper.

The key of the burette was operated carefully to make sure that one-drop of Acid falls into the conical flask at a time.

The conical flask was kept on a white paper.
to trace the color change of the solution.

At first the key of the burette was fully opened to let the solution fall free to make the bubbles go out.

On the basis of the result it was found that the balance is very much sensitive. So for this reason the result can be varied.

While measuring the lower meniscus of the burette an error may be happened for the parallax error. So concentration should be needed here.

Discussion:—

As KMnO₄ is not a primary standard substance so determination of the strength of it will be erroneous. Many factors are responsible for this error. These are stated below:

i. The solution must be strongly acidic in order to avoid other side reactions. Here sulphuric acid is normally used. Nitric acid is unsuitable since nitrate ion is a moderately strong oxidizing agent which may interfere with KMnO₄. If HCl
is used it may be oxidized to chlorine by permanganate. If chloride ion is present, high results are obtained because some permanganate is used up in oxidizing the HCl to eliminate the interference of chloride ions. The main reason for using acid in the titration is that it transforms oxalate to oxalic acid and this acid solution reduces the KMnO₄. Any side reaction may hamper the result. So only sulphuric acid should be used.

\[
2 \text{MnO}_4^- + 16 \text{H}^+ + 10 \text{Cl}^- \rightarrow 2 \text{Mn}^{2+} + 5 \text{Cl}_2 + 8 \text{H}_2\text{O}
\]

ii Reading might be wrong due to poor eye sight and non-vertical readings. The readings must be taken vertically considering the lower meniscus of the concave surface of the liquid.

iii If any air bubble enters the burette while pouring acid, air bubble should be removed by dropping out the solution forcibly until the air bubble is out of the burette. Or else, the presence of air bubble will hamper the reading of the volume.
iv While performing the experiment we should be careful so that misuse of solution can not happen. While taking KMnO$_4$ solution from the burette, one or two drops might fall on the body surface of the conical flask and might stick on it. These drops would not participate in the reaction but we might count these drops in out reading from the burette.

v After pouring any substance in the conical flask with the help of the pipette, if there is any liquid at the tip of the pipette, it should be ignored. But by no means it should be blown out.

vi Burette and pipette was first washed with distilled water then those were rinsed. It was necessary to perform to get accurate result.

vii During our experiment the burette was leaking, and as it was not noticed earlier so error might have occurred. When it came to our consideration, we did necessary things to tight it. At first for our callousness, the result might have deceived us.
In the KMnO₄ solution, there remains some impurities mainly MnO₂. Some Mn²⁺ are produced from MnO₂ which reacts with KMnO₄ and forms more MnO₂. This is known as auto decomposition. This will manipulate the result.

\[
2\text{MnO}_4^{-1} + 2\text{H}_2\text{O} + 3\text{Mn}^{2+} \rightarrow 5\text{MnO}_2 + 4\text{H}^+
\]

Even in the distilled water there remains some reducing agent which reacts with MnO₂ and produces more MnO₂ and more auto decomposition occurs. This is also responsible for erroneous result. To safeguard this re-distilled water from alkaline permanganate should be used.

\[
4\text{MnO}_4^{-1} + 2\text{H}_2\text{O} \text{(containing reducing agent)} \rightarrow 4\text{MnO}_2 + 3\text{O}_2 + 4\text{OH}^{-1}
\]

Even in the distilled water there remain some reducing agents which react with MnO₂ and produce more MnO₂ and more auto decomposition occurs. This is also responsible for erroneous result.

\[
4\text{MnO}_4^{-1} + 2\text{H}_2\text{O} \text{(containing reducing agent)} \rightarrow 4\text{MnO}_2 + 3\text{O}_2 + 4\text{OH}^{-1}
\]
XI) Permanganate solution should be added moderately and at the same time it has to be stirred to clear the solution constantly. But nonreactor permanganate should no way be allowed to accumulate in the solution because this may result in auto decomposition which will definitely manipulate the result.

Reference books:—

1) A Text Book of Quantitative analysis. - A.L.Vogel
2) Practical Chemistry - Huq and Jabber Main