ENGINEERING CHEMISTRY
LABORATORY MANUAL – I
As per ANNA University syllabus
(For First Semester B.E., / B. Tech.students
common to all branches)

G.SANTHI
R.SARAVANAN
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Acknowledgement

At first and foremost of all we thank GOD for completion of this lab manual
We wish to express our heartfelt thanks to our CMS Trust for having blessed this venture.

We are grateful to the Principal Dr.A.M.K.Poduval M.Sc,Engg.,Ph.D,FIE.
CMS College of Engineering and Technology for his constant support &guidance in bringing out this manual.
We are thankful to our colleagues for their suggestions for the preparation of this manual.

We are also thankful to our family members for their Cooperation.
We sincerely acknowledge the various digital resources used in this Laboratory Manual.
Preface

Its a great privilege in bringing out this manual for I semester B.E / B. Tech students as per prescribed syllabus of Anna university, Coimbatore.
The principles and procedures of each experiment have been presented in simple language which is easily perceived. Relevant tables are given wherever necessary. Molecular weights of some important substances are given.
List of chemicals required for each experiment with short procedures, special instructions and viva voce with Graph sheets are all given which will be helpful for students.
We advice the students to do practicals with interest and get full benefit out of this book. Maintain your observation book and record neatly. We wish you all success in your career. May God bless you.

G. Santhi
R. Saravanan
# CHEMISTRY LABORATORY MANUAL-I

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<td></td>
</tr>
</tbody>
</table>

Total Marks obtained

Internal marks awarded

Signature of staff incharge S
**DISODIUM SALT OF EDTA**

\[
\text{HOOCH}_2\text{C} \quad \text{CH}_2\text{COO}^-\text{Na}^+ \\
\text{\underline{N-CH}_2-\text{CH}_2-N} \\
\text{\underline{Na}^+ \text{OOCH}_2\text{C} \quad \text{CH}_2\text{COOH}}
\]

**STRUCTURE OF EDTA**

\[
\text{HOOCH}_2\text{C} \quad \text{CH}_2\text{COOH} \\
\text{\underline{N-CH}_2-\text{CH}_2-N} \\
\text{\underline{HOOCH}_2\text{C} / \quad \text{CH}_2\text{COOH}}
\]

**ESTIMATION OF HARDNESS OF WATER SAMPLE - EDTA METHOD**
**AIM:**

To determine the total, permanent and temporary hardness in the given water sample by EDTA method. A solution of standard hard water and EDTA are provided.

**PRINCIPLE:**

EDTA is ethylene diamine tetra acetic acid. Since pure EDTA is not dissolved in water, Disodium salt of EDTA is taken for this experiment. It is a complexing agent.

Actually, hard water contains Ca\(^{2+}\) / Mg\(^{2+}\) ions, during this experiment. EBT is added to hard water. This will from a weak complex and the reaction is as follows.

\[ \text{[Ca}^{2+} / \text{Mg}^{2+}] + \text{EBT} \xrightarrow{\text{Buffer, pH} 8.10} \text{[Ca}^{2+} / \text{Mg}^{2+} - \text{EBT}] \text{ complex} \]

Hard water  Indication  Wine - red Colour (weak)

When EDTA is added to this hard water (Complex), EBT is replaced by EDTA and a stable metal ion with EDTA complex is formed.

\[ \text{[Ca}^{2+} / \text{Mg}^{2+} - \text{EBT}] \text{ complex + EDTA } \xrightarrow{\text{pH} 8.10} \text{[Ca}^{2+} / \text{Mg}^{2+} - \text{EDTA}] \text{ Complex + EBT} \]

Wine red colour  steel blue colour.
TITRATION –I

STANDARDISATION OF EDTA
(Std Hard water Vs EDTA)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of Std.Hardwater (ml)</th>
<th>Burette readings</th>
<th>Volume of EDTA solution (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>EBT</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Concordant Value \( V_1 = \) __________ ml

**CALCULATION:**

Volume of Std.Hardwater \( = 20 \text{ ml} \)

Volume of EDTA consumed \( V_1 = \) ______ ml (Concordant Value)

1 ml of Std.Hardwater contains 1 mg of CaCO₃

20 ml of Std.Hardwater contains 20 mg of CaCO₃

But

20 ml of Std.Hardwater consumes \( V_1 \) ml of EDTA

(ie) \( V_1 \) ml of EDTA Solution = 20 mg of CaCO₃

1 ml of EDTA Solution \( = \) \( \frac{20 \text{ mg of CaCO}_3}{V_1} \)

\( = \) __________ mg of CaCO₃
**REAGENTS REQUIRED:**

- EDTA
- Std. Hard Water
- EBT (Solochrome black-T)
- Ammonium chloride – Ammonium hydroxide buffer (pH-10)
- Sodium hydroxide (4N)
- Sample and boiled water.

**PROCEDURE:**

**TITRATION - I**

**STANDARDISATION OF EDTA**

(Std. Hard water Vs EDTA)

The butter is washed well and then it is rinsed with EDTA solution. Now burette is filled with EDTA Solution. 20ml of std. Hard water is pipetted out into a clean conical flask. 5ml of amm. buffer and 2 drops of EBT indicator is added. This wine red coloured solution is then titrated against EDTA taken in the burette. The change of wine red colour to Steel blue is the end point. Repeat the titration to get the concordant (same) value. Let the volume of EDTA be $V_1$ ml.
# TITRATION – II

## ESTIMATION OF TOTAL HARDNESS
(Water Sample Vs Std. EDTA)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of water sample (ml)</th>
<th>Burette readings</th>
<th>Volume of EDTA solution (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>EBT</td>
</tr>
</tbody>
</table>

Concordant Value $V_2 = \underline{\text{ml}}$

**CALCULATION:**

Volume of water sample $= 20 \text{ ml}$

Volume of EDTA consumed $V_2 = \underline{\text{ml}}$ (Titre Value)

20 ml of given water sample consumes $V_2 \text{ ml}$ of EDTA

$V_2 = \frac{20 \times V_2}{V_1} \text{ mg of CaCO}_3$

1000 ml of given water sample contains $\frac{20 \times V_2 \times 1000}{V_1}$ mg of CaCO$_3$

$V_2 = \frac{V_2 \times 1000}{V_1} \text{ mg of CaCO}_3$

Total Hardness of given water sample $= \underline{\text{ppm}}$
**TITRATION - II**

**ESTIMATION OF TOTAL HARDNESS OF WATER SAMPLE**

*(Water Sample Vs EDTA)*

Pipette out 20ml of water sample into a clean conical flask. Add 5ml of buffer and 2drops of EBT to it. This wine red coloured solution is then titrated against EDTA taken in the burette. The end point is the colour change from wine red to steel blue. Repeat the titration to get the concordant value. Let the volume of EDTA consumed be V₂ ml.
ESTIMATION OF PERMANENT HARDNESS

[Water Sample (after boiling and filtering) Vs Std. EDTA]

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of boiled water (ml)</th>
<th>Burette readings</th>
<th>Volume of EDTA solution (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>EBT</td>
</tr>
</tbody>
</table>

Concordant Value $V_3 = \_\_\_\_\_\_\_\_\_ ml$

**CALCULATION:**

Volume of boiled water $= 20$ ml

Volume of EDTA consumed $V_3 = \_\_\_\_\_\_\_\_\_ ml$ (Titre Value)

20 ml of given water sample consumes $V_3$ ml of EDTA

(ie) 20 ml of given water sample contains $\frac{20 \times V_3}{V_1}$ mg of CaCO$_3$

1000 ml of given water sample contains $\frac{20 \times V_3 \times 1000}{V_1}$ mg of CaCO$_3$

(ie) $\frac{V_3}{V_1} \times 1000$ mg of CaCO$_3$

Permanent Hardness of given water sample = \_\_\_\_\_\_\_ ppm

**TITRATION - III**
ESTIMATION OF PERMANENT HARDNESS

100ml of given water sample is taken is a clean 250ml breaker and boiled for 10 - 15 minutes. It is then cooled and filtered. The filtrate is collected in a 100ml SMF and makes up to the mark. 20ml of this (made up) solution is pipetted out into a clean conical flask. Add 5ml of amm. buffer and 2 drops of EBT. This wine red coloured solution is titrated against EDTA taken in the burette. The end point is colour change from wine red to steel blue. Repeat the titration to get the concordant value. Let the volume of EDTA consumed be V₃ ml.

Temporary hardness can be calculated by using the equation,

Temporary hardness = Total Hardness - Permanent Hardness.

RESULT:
1. Amount of Total Hardness of given water sample = -------------- ppm
2. Amount of Permanent Hardness of given water sample =----------- ppm
3. Amount of Temporary Hardness of given water sample =---------- ppm

ESTIMATION OF TEMPORARY HARDNESS:

Temporary Hardness = TotalHardness - Permanent Hardness
INSTRUCTIONS TO STUDENTS

TITRATION - I

STANDARDISATION OF EDTA
• Fill the burette with EDTA
• Pipette out 20ml of standard water into a clean conical flask
• Add 5ml of freshly prepared ammonia buffer.
• Add 2-3 drops of EBT indicator
• Titrate the wine red solution with EDTA until steel blue colour appears (End Point)
• Repeat the titration to get concordant value.
• Let the volume of EDTA be \( V_1 \)ml.

**TITRATION - II**

**ESTIMATION OF TOTAL HARDNESS**

• Take EDTA in the burette
• Pipette out 20ml of sample water into a clean conical flask.
• Add 5ml of freshly prepared ammonia buffer
• Add 2-3 drops of EBT indicator
• Titrate the wine red solution with EDTA until steel blue colour appears (End Point)
• Repeat the titration to get concordant value.
• Let the volume of EDTA be \( V_2 \)ml

**TITRATION - III**

**ESTIMATION OF PERMANENT HARDNESS**

• Take EDTA in the burette
• Boil the water sample gently for 10 minutes

• Cool and filter
• Pipette out 20ml of sample water into a clean conical flask.
• Add 5ml of freshly prepared ammonia buffer
• Add 2-3 drops of EBT indicator
• Titrate the wine red solution with EDTA until steel blue colour appears
• Repeat the titration to get concordant value.
• Let the volume of EDTA be $V_3$ml

**VIVA VOCE:**

1. Water which contains $\text{Ca}^{2+}$ & $\text{Mg}^{2+}$ salts (i.e. Carbonate, bicarbonates chlorides and sulphates) are called Hard water.
2. ii) Types of Hardness: Temporary and Permanent Hardness
   Temporary Hardness: Presence of carbonates & and bicarbonates of $\text{Ca}^{2+}/\text{Mg}^{2+}$.
   Permanent Hardness: Presence of Chlorides and sulphates of $\text{Ca}^{2+}/\text{Mg}^{2+}$.
   Temporary Hardness can be removed by boiling the water. Permanent hardness can be removed by the softening (conditioning) process. There are two types of softening methods namely.
   a) External treatment (zeotite (or) Permulit process and ion-exchange process)
   b) Internal treatment (Colloidal, Phosphate, Carbonate and Calgon Conditioning)
   
   Total Hardness = Temporary Hardness - Permanent Hardness

3. EDTA: Ethylene Diamine Tetra Acetic acid

4. Units of Hardness
   - ppm, mg/L, $^0\text{Fr}$, $^0\text{Cl}$
   - $1 \text{ ppm} = 1\text{mg/L} = 0.1^0\text{Fr} = 0.07^0\text{Cl}$

5. Structure of EDTA

\[
\begin{align*}
\text{HooC}H_2C & \quad \text{N-} \quad \text{CH}_2 - \quad \text{CH}_2 - \quad \text{N} \\
\text{HooC}H_2C & \quad \text{CH}_2\text{CooH} \\
\text{Disodium Salt of EDTA} & \\
^+ \text{Na} & \quad \text{ooC}H_2C \\
& \quad \text{CH}_2\text{CooH}
\end{align*}
\]
6. Buffer is a solution used to maintain the pH range of a reaction. Buffer can be classified into two types namely Acidic & Basic

Example for acidic buffer: CH₃COOH / CH₃CooNa (Acetic acid/Sodium acetate)

Example for basic buffer: NH₄ OH/NH₄Cl

7. Reason for wine red colour

\[
\text{Ca}^{2+} / \text{Mg}^{2+} + \text{EBT} \rightarrow \text{[Ca}^{2+} / \text{Mg}^{2+} - \text{EBT]} \]

Complex

Hard water wine red colour

8. Reason for steel blue colour

\[
\text{[Ca}^{2+} / \text{Mg}^{2+} - \text{EBT]} \text{ complex} + \text{EDTA} \rightarrow \text{[Ca}^{2+}/\text{Mg}^{2+} - \text{EDTA]} + \text{EBT}
\]

(Wine Red Colour) (Steel Blue Colour)

9. Expansion of pH is potential Hydrogen

\[
\text{pH} = - \log [H^+]\]

10. pH Scale:

- if pH = 7 the solution is neutral (for pure water)
- pH = 0 to 7 (for acids)
- pH = 7 to 14 (for bases)
**ALKALINITY CONDITIONS**

[Using Phenolphthalein [P] and Methyl Orange [M] values]

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Result of Titration [P] and [M]</th>
<th>Alkaline causing ions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[P] = 0</td>
<td>OH⁻</td>
</tr>
<tr>
<td>1</td>
<td>[P] = 0</td>
<td>Nil</td>
</tr>
<tr>
<td>2</td>
<td>[P] = [M]</td>
<td>[P] or[M]</td>
</tr>
<tr>
<td>3</td>
<td>[P] = ( \frac{1}{2} [M] )</td>
<td>Nil</td>
</tr>
<tr>
<td>4</td>
<td>[P] &gt; ( \frac{1}{2} [M] )</td>
<td>2[P] [M]</td>
</tr>
<tr>
<td>5</td>
<td>[P] &lt; ( \frac{1}{2} [M] )</td>
<td>Nil</td>
</tr>
</tbody>
</table>

Table – 1
DETERMINATION OF ALKALINITY OF WATER SAMPLE

Expt.No. Date:

AIM:
To determine the type and amount of alkalinity present in the given sample of water. A standard NaOH solution of strength \( \text{--------} \) N and HCl solutions are provided.

PRINCIPLE:

Alkalinity of water is due to the presence of hydroxide (OH\(^-\)) carbonate \((CO_{3}^{2-})\) and bicarbonate \((HCO_{3}^{-})\) ions. There are five alkalinity conditions are possible in a water sample. These conditions can be estimated by titrating with standard acid using phenolphthalein (P) and methyl orange (M) indicators.

(i) Phenolphthalein End Point:

Hydroxide (OH\(^-\)) ion alkalinity is completely neutralized and carbonate \((CO_{3}^{2-})\) alkalinity is partially neutralized during phenolphthalein end point.

\[
\text{OH}^- + \text{H}^+ \rightarrow \text{H}_2\text{O}
\]

\[
CO_{3}^{2-} + \text{H}^+ \rightarrow HCO_{3}^{-}
\]
(ii) **Methyl Orange End point:**

Bicarbonate ion neutralize during methyl orange end point only

\[
HCO_3^- + H^+ \rightarrow CO_2 + H_2O
\]

From the two titre values the different alkalinities are calculated. It is given in the table (1)

If, \([P] = 0\); only bicarbonate ion is present

\([P] = M\); only hydroxide ion is present

\([P] = 1/2M\); only carbonate ion is present.

\([P] < 1/2M\); Carbonate and bicarbonate ions are present.

\([P] > 1/2M\); hydroxide & carbonate ions are present.

\[CO_3^{2-} = 2P\]

\[HCO_3^- = M-2P\]

\[CO_3^{2-} = 2(M-P)\]

\[OH^- = 2P-M\]

Alkalinity values are expressed in term of milligram per litre as calcium carbonate.

**REAGENTS REQUIRED:**

Sulphuric acid (0.02M), Sulphuric acid (0.1M), (or) hydrochloric aid (0.02N), Hydrochloric acid (0.1N), Phenolphthalein (0.5%), Methyl orange (0.5%)

**TITRATION –I**

**STANDARDISATION OF HCl**
( Std.NaOH Vs HCl )

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of NaOH (ml)</th>
<th>Burette readings</th>
<th>Volume of HCl (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>Phenolphthalein</td>
</tr>
</tbody>
</table>

Concordant Value = ____________ ml

CALCULATION:

\[
\begin{align*}
\text{Volume of NaOH Solution} & : V_1 = 20 \text{ ml} \\
\text{Strength of NaOH Solution} & : N_1 = \text{_______ N (given)} \\
\text{Volume of HCl} & : V_2 = \text{_______ ml (Concordant Value)} \\
\text{Strength of HCl} & : N_2 = \frac{V_1 \times N_1}{V_2} \\
\end{align*}
\]

\[
\begin{align*}
20 & \times \text{_________} \\
\end{align*}
\]

\[
\begin{align*}
\text{________} \\
\end{align*}
\]

\[
\begin{align*}
\text{________} \\
\end{align*}
\]

\[
\begin{align*}
\text{Strength of HCl} & = \text{_______ N} \\
\end{align*}
\]

**PROCEDURE:**
TITRATION - I

STANDARDISATION OF HCl

(Std. NaOH Vₙ HCl)

Burette is washed with water and rinsed with HCl. Then it is filled with HCl. Pipette out 20ml of Std. NaOH in a clean conical flask. Add 1-2 drops of phenolphthalein as indicator. This pink coloured solution is titrated with HCl taken in the burette; the end point is the disappearance of pink colour. Repeat the titration to get the concordant value.

TITRATION –II

ESTIMATION OF ALKALINITY
(Water Sample Vs Std. HCl)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of Water sample (ml)</th>
<th>Burette readings</th>
<th>Volume of HCl (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>P</td>
</tr>
<tr>
<td></td>
<td></td>
<td>P</td>
<td>M</td>
<td></td>
</tr>
</tbody>
</table>

Concordant Value ; P = __________ml

M = __________ml

\[ \frac{1}{2} M = \frac{1}{2} \times \text{nil} \]

**CALCULATION:**
After looking the P and M Values, it will satisfy any one of that five alkalinity conditions.

**For Example:**

Alkalinity due to OH\(^{-}\) ions = nil

If \( P < \frac{1}{2} M \)

**TITRATION - II**

**ESTIMATION OF ALKALINITY**
Burette is filled with HCl. Pipette out 20ml of water sample into a clean conical flask. Add 1-2 drops of phenolphthalein indicator. This pink coloured solution is titrated against HCl taken in the burette till the pink colour disappears. Note this point as (P).

To the same colourless solution add 1-2 drops of methyl orange indicator. This yellow coloured solution is titrated against HCl from the point (P). The end point is colour change from yellow to red orange. Note this point as (M).

Repeat the titrations to get the concordant value.

**RESULT:**

(i) Alkalinity due to \(OH^-\) ion = __________ ppm.

(ii) Alkalinity due to \(CO_3^{2-}\) ion = __________ ppm.

(iii) Alkalinity due to \(HCO_3^-\) ion = __________ ppm.

**\(CO_3^{2-}\) ion alkalinity:**

Volume of HCl \(V_1 = 2P\)

\[= 2 \times \text{---} \]

\[= \text{---} \text{ml}\]
Strength of HCl \( N_1 \) = __________ N (I calculation)

Volume of water sample \( V_2 \) = 20 ml

Strength of water sample \( N_2 \) = \( \frac{V_1 N_1}{V_2} \)

Strength of Water Sample = __________ N

Amount of \( \text{CO}_3^{2-} \) ion present in

1 lit of water sample in terms of \( \text{CaCO}_3 \) equivalent

\[ = \text{Eq.Wt of } \text{CaCO}_3 \times \text{strength of water sample} \]

\[ = 50 \times 1000 \text{ ppm} \times \text{__________ N} \]

Alkalinity due to \( \text{CO}_3^{2-} \) ions = __________ ppm

\( \text{HCO}_3^- \) - ion alkalinity:

Volume of HCl \( V_1 = [M] - 2 [P] \)

\[ = \text{___________ ml} \]

Strength of HCl \( N_1 \) = __________ N (I calculation)

Volume of water sample \( V_2 \) = 20 ml

Strength of water sample \( N_2 \) = \( \frac{V_1 N_1}{V_2} \)

Strength of Water Sample = __________ N

Amount of \( \text{HCO}_3^- \) ion present in

1 lit of water sample in terms of \( \text{CaCO}_3 \) equivalent

\[ = \text{Eq.Wt of } \text{CaCO}_3 \times \text{strength of water sample} \]

\[ = 50 \times 1000 \text{ ppm} \times \text{__________ N} \]

\( \text{HCO}_3^- \) ions Alkalinity = __________ ppm

**Instructions to Candidates:**

**Standardisation of Acid:**

- Fill burette with acid
- Pipette out 20ml of NaOH in clean conical flask
- Add 2 drops of phenolphthalein indicator
- Titrate the yellow solution against acid till colour changes to orange - pink.

**Estimation of alkalinity:**
- Fill burette with standardized acid
- Pipette out 20ml of sample water in clean conical flask
- Add one drop of 0.1N thio to remove the free residual chlorine if present.
- Add 2 drops of phenolphthalein indicator
- Titrate the pink solution against acid till colour changes from pink to colourless
- Note the burette reading
- To the same sample water add 2drops of methyl orange indicator.
- Resume the titration by titrating the yellow orange solution against acid until orange - pink colour is noted (end point).
- The total volume of acid added is noted.

**Viva Voce:**

i) Alkalinity is due to the presence of hydroxyl (OH⁻), Carbonate (\(CO_3^{2-}\)) and bicarbonates (\(HCO_3^-\)) ions.

ii) There is no water which contains all the three ions. Because any two will combine to from the other. One therefore for any water either two (or) only one alkaline ions are present.

iii) In the second titration two indicators are used because bicarbonate (\(HCO_3^-\)) ions shows the end point only in the methyl orange indicator.
## Short Procedure

<table>
<thead>
<tr>
<th>Burette solution</th>
<th>Standarisation of HCl Std (NaOH $V_S$ HCl)</th>
<th>Estimation of Alkalinity (Water sample $V_S$ HCl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pipette solution</td>
<td>HCl Std NaOH, 20ml</td>
<td>HCl Water sample, 20ml</td>
</tr>
<tr>
<td>Indicator</td>
<td>Phenolphthalein</td>
<td>i) Phenolphthalein</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ii) Methyl orange</td>
</tr>
<tr>
<td>End point</td>
<td>Disappearance of pink colour</td>
<td>i) Disappearance of pink colour.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ii) Appearance of orange-pink colour.</td>
</tr>
</tbody>
</table>
ESTIMATION OF DISSOLVED OXYGEN (DO) IN WATER SAMPLE (WINKLER’S METHOD)

Expt.No ........................................ Date:

AIM:
To determine the amount of dissolved oxygen (D.O) in tap water by Winkler’s method. A standard solution of K₂Cr₂O₇ of strength -------- N, sodium thio sulphate are provided.

PRINCIPLE:
Oxygen is dissolved in pure water to a definite proportion. The oxygen content may decrease because of the presence of organic impurities (because of aerobic oxidation). The oxygen content in pure water may be 5-6 mgs/ lit at a temperature range of 25°C - 35°C.

Water sample is collected carefully in a stoppered flask. Manganese sulphate solution in presence of alkali iodide is added to water in an iodine flask. The following reaction occurs.

\[ \text{Mn}^{2+} + 2\text{OH}^- \rightarrow \text{Mn(OH)}_2 \downarrow \]  
(White ppt)

\[ \text{Mn(OH)}_2 + \frac{1}{2}\text{O}_2 \rightarrow \text{MnO(OH)}_2 \downarrow \]  
Yellowish brown ppt

Dissolved Oxygen in water in utilized for the second reaction. After some time con. H₂SO₄ and KI are added and the reaction is

\[ \text{MnO(OH)}_2 + 2\text{I}^- + 4\text{H}^+ \rightarrow \text{I}_2 + \text{Mn}^{2+} + 3\text{H}_2\text{O} \]

The known value of this solution is titrated with Na₂S₂O₃ solution.
$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$
Freshly prepared starch solution is added to the conical flask when it is nearing the end point. The end point is the disappearance of blue colour.

Sodium thiosuphate can be standardized using potassium dichromate liberates I₂ from KI in acidic medium the reaction is,

\[
\text{Cr}_2\text{O}_7^{2-} + 14 \text{H}^+ + 6\text{I}^- \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O} + 3 \text{I}_2
\]

Orange \quad \text{Pale green}

The liberated iodine is titrated against thiosulphate. Starch is added near the end point, reason is Iodine release is difficult. The end point is disappearance of blue colour leaving behind pale green colour.

**REAGENTS REQUIRED:**

Sodium thio sulphate (0.01N), Potassium Iodide 10%, Potassium dichromate (0.01N), Starch, Dilute and concentrated sulphuric acid, Manganous Sulphate, Alkaline Potassium Iodide.
## STANDARDISATION OF SODIUM THIOSULPHATE

(Std.$K_2Cr_2O_7$ Vs Sodium thiosulphate)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of $K_2Cr_2O_7$ (ml)</th>
<th>Burette readings</th>
<th>Volume of Sodium thiosulphate (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Starch</td>
</tr>
</tbody>
</table>

Concordant Value = ___________ ml

**CALCULATION:**

Volume of $K_2Cr_2O_7$ Solution $V_1 = 20$ ml

Strength of $K_2Cr_2O_7$ Solution $N_1 = \text{_________ N (given)}$

Volume of Sodium thiosulphate Solution $V_2 = \text{_______ ml (ConcordantValue)}$

Strength of Sodium thiosulphate Solution $N_2 = \frac{V_1 \cdot N_1}{V_2}$

\[ 20 \times \text{_______} = \text{______________________} \]

Strength of Sodium thiosulphate Solution $= \text{_________ N} $
PROCEDURE:

TITRATION - I

STANDARDISATION OF SODIUM THIO SULPHATE

Burette is filled with sodium thiosulphate. 20ml of standard K₂Cr₂O₇ is pipetted out into a clean conical flask. To this 10ml of sulphuric acid and 10ml of 5% Potassium Iodide are added. This is titrated against sodium thiosulphate solution. When the solution becomes straw yellow colour, starch indicator is added and then titration is continued.

The end point is the colour change from blue to light green colour. The titration is repeated to get concordant value.

TITRATION –II
ESTIMATION OF DISSOLVED OXYGEN  
(Tap Water Vs Sodium thiosulphate)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of Tap water (ml)</th>
<th>Burette readings</th>
<th>Volume of Sodium thiosulphate (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>Starch</td>
</tr>
</tbody>
</table>

Concordant Value = ___________ ml

**CALCULATION:**

Volume of Sodium thiosulphate Solution  \( V_1 = _____ ml \)

Strength of Sodium thiosulphate Solution  \( N_1 = ______ N \) (1 calculation)

Volume of Water sample  \( V_2 = 100 \) ml

Strength of Water sample (or)

Strength of Dissolved oxygen in water  \( N_2 = _____ \) ?

\[
= \frac{V_1 \times N_1}{V_2}
\]

\[
____ ml \times _____
\]

\[
= \frac{_____}{100}
\]

\[
= \text{---------- N}
\]

**PROCEDURE:**

**TITRATION - II**
ESTIMATION OF DISSOLVED OXYGEN

250ml iodine flask is filled with tap water upto the neck. Add 2ml of alkali iodide (NaOH & KI) and 2ml of manganous sulphate solution are added and stoppered. Iodine flask is shaken and kept for about 20 minutes for setting the precipitate.

Then a few ml of con.H₂SO₄ are added to get a clear yellow solution due to iodine liberation.

100ml of this solution is measured in a measuring jar and transferred to a conical flask. This is titrated with sodium thiosulphate in the burette. Starch is added nearing the end point. End point is the disappearance of blue colour. Titre value is noted.

Eq.wt of Oxygen = 8

RESULT
Amount of DO present in tapwater = -------------- mg / lit.

Amount of Dissolved oxygen in 1 lit of Tapwater

= Eq.Wt of O₂ X 1000 X Normality

= 8 x 1000 x -------- N

= _____________ mgs / lit

INSTRUCTIONS TO STUDENT
TITRATION - I
Standardisation of Sodium thio sulphate:
- Fill burette with sodium thio sulphate
- Pipette out 20ml of potassium dichromate in a clean conical flask
- Add one test tube sulphuric acid (4N)
- Add one test tube 10% KI solution.
- Titrate this reddish brown solution against thio until a pale yellow colour is obtained.
- Add one ml of freshly prepared starch indicator.
- Continue the titration against the same thio until blue colour disappears.
- Note the end point.
- Repeat the titration for concordant value.

**TITRATION - II**

**Estimation of dissolved oxygen:**
- Take 250ml of sample water in a bottle.
- Add 2ml of saturated manganous sulphate
- Add 2ml of alkaline KI solution.
- Stopper the bottle and shake well.
- Allow 20 minutes for the precipitate to settle down.
- Dissolve the precipitate using 2ml of conc.sulphuric acid
- Titrate this against sodium thiosulphate solution using starch as indicator.
- Disappearance of blue colour (end point)
- Repeat the titration for concordant value.

**Viva Voce Questions:**
1. Determination method: Winkler’s method.
2. The other names of sodium thiosulphate is Hypo (or) thio.
3. Air is dissolved in water in a definite quantity the oxygen content is
decrease reason is presence of organic impurities.
4. Starch should not be added at beginning. This will form a blue
coloured precipitate adsorption complex, from which I₂ release is
difficult.
5. Equivalent weight of oxygen = 8
6. During this titration iodine gas is liberated. So iodine flask is used.
Hence this experiment is called is an Iodimetric experiment.
7. Alkali-Iodine solution contains NaOH and KI.

**SHORT PROCEDURE**

<table>
<thead>
<tr>
<th>Burette Solution</th>
<th>Standardisation of Thiosulphate Std K₂Cr₂O₇ Vₛ Na₂S₂O₃</th>
<th>Estimate of Dissolved oxygen water sample Vₛ Na₂S₂O₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium thiosulphate</td>
<td>Sodium thiosulphate</td>
<td>S</td>
</tr>
<tr>
<td>Pipette Solution</td>
<td>Potassium dichromate</td>
<td>Water Sample (20ml)</td>
</tr>
<tr>
<td>Added Solution</td>
<td>5%KI (10ml) + dil H₂SO₄ (10ml)</td>
<td>Alkali Iodide (2ml) + MnSO₄ (2ml)</td>
</tr>
<tr>
<td>Indicator</td>
<td>Starch</td>
<td>Starch</td>
</tr>
<tr>
<td>End Point</td>
<td>Blue colour change to pale green colour</td>
<td>Blue colour change to pale green colour.</td>
</tr>
</tbody>
</table>
DETERMINATION OF CHLORIDE CONTENT OF WATER
SAMPLE - ARGENTOMETRIC METHOD

Expt No. Date:

AIM

Determine the amount of chloride ion present in the given water sample by Argento metric method. You are provided with a standard solution of NaCl of strength ------------N and a link solution AgNO₃.

PRINCIPLE

Generally natural water contains chloride ions with Na, K, Ca & Mg. This amount can be determined by argento metric method. Here chloride ion solution is titrated with AgNO₃, Pot. Chromate (K₂ CrO₄) as indicator. The reaction is,

\[ \text{AgNO}_3 + \text{NaCl} \rightarrow \text{AgCl} \downarrow + \text{NaNO}_3 \]
\[ (\text{Burette}) \quad \text{(in water)} \quad \text{(white ppt)} \]
\[ \text{AgNO}_3 + \text{K}_2\text{CrO}_4 \rightarrow \text{Ag Cr O}_4 + 2\text{K NO}_3 \]
\[ (\text{indicator}) \quad \text{(reddish yellow)} \quad \text{brown}) \]

It is a precipitation titration, initially, AgNO₃ is added to the NaCl solution, a white precipitate of AgCl is formed. To this indicator K₂CrO₄ is added, it is then titrated with AgNO₃. Finally an end point of reddish brown colour is formed due to the formation of silver chromate.

This method is also known as Mohr’s titration
**TITRATION – I**

**STANDARDISATION OF AgNO₃**

(Std. NaCl Vs AgNO₃)

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Volume of NaCl (ml)</th>
<th>Burette readings</th>
<th>Volume of AgNO₃ (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td></td>
</tr>
</tbody>
</table>

Concordant Value = ____________ ml

**CALCULATION:**

Volume of NaCl Solution \( V₁ = 20 \text{ ml} \)

Strength of NaCl Solution \( N₁ = \text{__________ N (given)} \)

Volume of AgNO₃ Solution \( V₂ = \text{__________ ml} \)

(Concordant Value)

Strength of AgNO₃ Solution \( N₂ = \text{__________ N(from Titration - I)} \)

\[
N₂ = \frac{V₁ \cdot N₁}{V₂} \\
= \frac{20 \times \text{_______}}{\text{_______}} \\
= \text{________________} \\
\]

Strength of AgNO₃ Solution = ____________ N
REAGENTS REQUIRED

- Sodium chloride (0.1N)
- Sodium chloride (0.02N)
- Potassium chromate indicator (1%)
- Silver nitrate solution (0.02N)

PROCEDURE

TITRATION - I

STANDARDISATION OF AgNO₃
(AgNO₃ V₅ Std. NaCl )

Burette is filled with AgNO₃ solution, pipette out 20ml of std. NaCl solution. To this 1ml of K₂CrO₄ is added as an indicator. It is then titrated against AgNO₃ taken in the burette. The end point is colour change from yellow to permanent reddish brown, followed by the coagulation of white precipitate. Repeat the titrations to get the concordant value.
## TITRATION – II
### ESTIMATION OF CHLORIDE ION
(Water Sample Vs Std . AgNO₃)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of Water Sample (ml)</th>
<th>Burette readings</th>
<th>Volume of AgNO₃ (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>K₂CrO₄</td>
</tr>
</tbody>
</table>

**CALCULATION:**

- Volume of Water Sample \( V₁ = 100 \) ml
- Strength of Water Sample \( N₁ = \) _______ N
- Volume of AgNO₃ Solution \( V₂ = \) _______ ml (Concordant Value)
- Strength of AgNO₃ Solution \( N₂ = \) _______ N (from Titration I)

\[
N₁ = \frac{V₂ \times N₂}{V₁} \\
\]

\[
\text{Strength of Water Sample} = \text{______ N}
\]

Amount of Chloride ion present in

1 lit of water sample \( = \text{Eq.Wt x strength of water sample} \)

\[
= 35.45 \times \text{_______} \\
= \text{_______ g}
\]
TITRATION - II

ESTIMATION OF CHLORIDE ION IN WATER SAMPLE

(Water sample \( V_s \) AgNO\(_3\))

Burette is filled with AgNO\(_3\). 100ml of water sample is measured out into a clean conical flask. To this 1ml of K\(_2\)CrO\(_4\) is added as an indicator. It is then titrated against AgNO\(_3\) in burette. The end point is colour change from yellow to permanent reddish brown followed by the coagulation of white precipitate. Repeat the titrations to get the concordant value.

Equivalent weight of chloride ion = 35.45

RESULT:

Amount of chloride ion present in the given water sample = \(---------\)g
INSTRUCTION TO CANDIDATES

**STANDARDISATION OF SILVER NITRATE**

- Fill the burette with silver nitrate
- Pipette out 20ml of sodium chloride solution in clean conical flask
- Add 1ml of potassium chromate indicator
- Titrate the yellow solution against silver nitrate till colour change to reddish brown.
- Note the burette reading
- Repeat the titration for concordant value.

**ESTIMATION OF CHLORIDE**

- Fill burette with standardized silver nitrate solution.
- Pipette out 50ml of sample water in clean conical flask
- Add 1ml of potassium chromate indicator.
- Titrate the yellow solution against silver nitrate till colour changes to reddish brown (end point).
- Note the burette reading S
- Repeat the titration for concordant value.

**VIVA VOCE:**

1. Equivalent weight of chloride ion-35.45
2. Generally water contains Chloride ions due to the presence of NaCl, KCl, CaCl₂, MgCl₂.
3. The concentration of Chloride ions more than 250ppm is not desirable for drinking purpose.
4. The other name for this method is Mohr’s method.
5. Addition of silver nitrate from the burette to the water sample produces white precipitate due to the formation of AgCl

\[
\text{AgNO}_3 + \text{Cl}^- \rightarrow \text{AgCl} \downarrow + \text{NO}_3^-
\]

(Water sample ion)

6. The reddish brown colour is due to the formation silner chromate precipitate.

\[
2\text{AgNO}_3 + \text{K}_2\text{CrO}_4 \rightarrow \text{Ag}_2\text{CrO}_4 \downarrow + 2\text{KNO}_3
\]

(yellow) (reddish brown ppt)
ESTIMATION OF COPPER IN BRASS BY IODOMETRY

Expt No.  Date:

AIM

To estimate the amount of Copper present in the brass solution. A standard solution of Pot. dichromate of strength ----------- N and sodium thiosulphate as link solution are provided.

PRINCIPLE

Alloys are the mixture of two or more elements. Brass in an alloy of Copper, iron, Zinc and Tin. It has the percentage of copper 55%.

1g of brass is dissolved in minimum nitric acid by careful heating to remove nitrogen oxides. The residual oxides of nitrogen are removed by adding 10ml of con. H₂SO₄ and heating the contents for evaporation. It is then cooled and 20ml of distilled water is added. Boil the solution for few more minutes and cool it.

Liquid ammonia is added with a constant stirring and deep blue precipitate is obtained. It is then neutralized by adding 6N H₂SO₄. Then add about 5ml of orthophosphoric acid which complexes the ion. Finally the solution is transferred into 100ml SMF. This a brass solution. It is titrated against Na₂S₂O₃ using KI and starch as indicator. The reaction are,

\[ 2Cu^{2+} + 4I^- \rightarrow Cu_2I_2 + I_2 \]

\[ I_2 + 2S_2O_3^{2-} \rightarrow S_2O_4^{2-} + 2I^- \]

Initially sodium thiosulphate is standardized using std. K₂Cr₂O₇ is acid medium and by adding KI solution. Here Iodine gas is liberalized by dichromate. This liberated iodine is titrated against Na₂S₂O₃ using starch as an indicator.

\[ Cr_2O_7^{2-} + 14H^+ + 6I^- \rightarrow 2Cr^{3+} + 7H_2O + 3I_2 \]

(Chromous ion-Orange) (Chromic ion-green)
# TITRATION –I

## STANDARDISATION OF SODIUM THIOSULPHATE

(Sodium thiosulphate Vs Std.K₂Cr₂O₇)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of K₂Cr₂O₇ (ml)</th>
<th>Burette readings</th>
<th>Volume of Sodium thiosulphate (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>Starch</td>
</tr>
</tbody>
</table>

Concordant Value = _____________ ml

**CALCULATION:**

- **Volume of K₂Cr₂O₇ Solution** \( V_1 = 20 \text{ ml} \)
- **Strength of K₂Cr₂O₇ Solution** \( N_1 = \square \text{ N} \) (given)
- **Volume of Sodium thiosulphate Solution** \( V_2 = \square \text{ ml} \)

(Concordant Value)

- **Strength of Sodium thiosulphate Solution** \( N_2 = \frac{V_1 N_1}{V_2} = \frac{20 \times \square}{\square} = \square \)

Strength of Sodium thiosulphate Solution = \( \square \text{ N} \)
**CHEMICALS REQUIRED**

1. Sodium thiosulphate 0.1N
2. Sulphuric acid 4N
3. Potassium dichromate 0.1N
4. Starch solution 1N.
5. Brass solution
6. Potassium Iodide 10N.
7. Acetic acid
8. Brass solution
9. Ammonium hydroxide.

**PROCEDURE**

**TITRATION – I**

**STANDARDISATION OF SODIUM THIOSULPHATE**

\((\text{Na}_2\text{S}_2\text{O}_3 \text{ V}_5 \text{ Std. K}_2\text{Cr}_2\text{O}_7)\)

Burette is filled with thiosulphate solution. Pipette out 20ml of std. pot.dichromate into a clean concial flask. To this 10ml of dil. H\(_2\)SO\(_4\) and 15ml of 10% KI are added. the liberated iodine is titrated against sodium thiosulphate when the colour becomes straw yellow, 1ml of freshly prepared starch is added as indicator. Continue the titration, the endpoint is the appearance of green colour. Repeat the titrations to get the concordant value.
TITRATION – II
ESTIMATION OF COPPER IN BRASS
(Sodium thiosulphate Vs Brass Solution)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Volume of Brass Solution (ml)</th>
<th>Burette readings</th>
<th>Volume of Sodium thiosulphate (ml)</th>
<th>Indicator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial (ml)</td>
<td>Final (ml)</td>
<td>Starch</td>
</tr>
</tbody>
</table>

Concordant Value = ____________ ml

**CALCULATION:**

Volume of Sodium thiosulphate Solution \( V_1 = 20 \text{ ml} \)

Strength of Sodium thiosulphate Solution \( N_1 = \text{__________ N (given)} \)

Volume of Brass Solution \( V_2 = \text{__________ ml (Concordant Value)} \)

Strength of Brass Solution \( N_2 = \frac{V_1 - N_1}{V_2} \)

\[
N = \frac{20 \times \text{__________}}{\text{__________}}
\]

Strength of Brass Solution = ____________ N

Amount of Copper present in given Brass Solution

\[
= \text{Eq.Wt} \times \text{strength of Brass Solution}
= 63.54 \times \text{__________}
= \text{__________ g}
\]
ESTIMATION OF COPPER IN BRASS

Preparation of Brass Solution

Exactly 1g of brass is weighted and 10ml of 6N Nitric acid is added to it. It is then heated gently till the brass is completely dissolved. Excess of nitrate and oxides of nitrogen is removed by adding 10ml of Con. H₂SO₄, heated gently. It is then cooled and 20ml of distilled water is added carefully from the sides of the breaker boil it for few minutes. Liquid ammonia is added with a constant stirring until the blue precipitate just disappears. Finally 5ml of ortho phosphoric acid is added to complexes the iron is the brass. This solution is transferred into a 100ml SMF and made upto the mark with distilled water. Thus copper sulphate solution (Brass solution) is obtained from the brass.

TITRATION – II

Burette is filled with sodium thiosulphate solution. Pipette out 20ml of made up brass solution into a clean conical flask. Few drops of NH₄OH solution is added drop by drop till faint precipitate is obtained. It is then neutralized by adding 2ml of dil. CH₃COOH to get a clear solution then 10ml 10% KI is added. Titrate the solution with sodium thiosulphate till straw yellow colour appears. Now 1ml of starch is added as indicator. Continue the titration till ivory colour (or) pale white colour is obtained. Repeat the titrations to get the concordant value.

Eq.wt. of Copper = 63.54

RESULT

The amount of Copper present is given brass = -------------- g

INSTRUCTIONS
**TITRATION – I**
- Fill the burette with sodium thiosulphate
- Pipette out 20ml of standard pot. dichromate
- Add 10ml of dil.H₂SO₄ and 15ml of 10% KI.
- Titrate librated iodine against sodium thiosulphate until straw yellow appears.
- Add 1ml of starch indicator and continue
- Repeat the titration to get concordant value

**TITRATION – II**
- Fill the burette with sodium thiosulphate
- Pipette out 20ml of brass solution
- Add two drops of NH₄OH, 2ml of dil.CH₃COOH and 10ml of 10%KI
- Titrate the solution with sodium thiosulphate till straw yellow colour appears.
- Add 1ml of starch indicator and continue the titration until place white colour appears.

**SIMPLE PROCEDURE:**

**TITRATION – I**

<table>
<thead>
<tr>
<th>Burette Solution</th>
<th>Sodium thiosulphate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pipette Solution</td>
<td>Std. Potassium dichromate</td>
</tr>
<tr>
<td>Additional Solution</td>
<td>10ml of dil.H₂SO₄+15ml of 10%KI.</td>
</tr>
<tr>
<td>Indicator</td>
<td>Starch</td>
</tr>
<tr>
<td>End point</td>
<td>Appearance of green colour.</td>
</tr>
</tbody>
</table>

**TITRATION – II**

| Burette Solution | Sodium thiosulphate |
Pipette Solution - Brass solution
Additional Solution - Few drops NH₄OH+2ml of dil.CH₃CooH+10ml 10%KI
Indicator - 1ml Starch
End point - Appearance of pale white colour.

**VIVA VOCE**

1. Brass contains Cu-55%, Zn-33%, Fe, Mn, Pb, Sn.
2. This experiment is called as Iodometric titration because during the reaction Iodine gas is liberating
   \[2\text{Cu}^{2+} + 4\text{I} \rightarrow 2\text{CuI} + \text{I}_2\]
3. Sodium thiosulphate also called as thio (or) hypo
4. During the end point Cr⁶⁺ is converted to Cr³⁺
Table I
Example for preparation of various concentrated Polymer Solution

<table>
<thead>
<tr>
<th>Sl .No</th>
<th>Volume of 5% Polymer Solution (Stock solution) ($V_1$ ml)</th>
<th>Volume of water (ml)</th>
<th>Concentration C (%) ($N_2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>5</td>
<td>45</td>
<td>($V_1 N_1 = V_2 N_2$)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>($5 \times 5) / 50 = 0.5$</td>
</tr>
<tr>
<td>II</td>
<td>10</td>
<td>40</td>
<td>($5 \times 10) / 50 = 1.0$</td>
</tr>
<tr>
<td>III</td>
<td>15</td>
<td>35</td>
<td>($5 \times 15) / 50 = 1.5$</td>
</tr>
<tr>
<td>IV</td>
<td>20</td>
<td>30</td>
<td>($5 \times 20) / 50 = 2.0$</td>
</tr>
<tr>
<td>V</td>
<td>25</td>
<td>25</td>
<td>($5 \times 25) / 50 = 2.5$</td>
</tr>
</tbody>
</table>
DETERMINATION OF MOLECULAR WEIGHT OF
A POLYMER BY VISCOMETRY

Expt. No. Date:

AIM
To determine the molecular weight of a given polymer using Ostwald’s viscometer, a polymer solution is provided.

PRINCIPLE
The strength of a polymer is depend on its molecular weight. Increase in molecular weight, the strength of polymer increases. This can be determined by measuring the intrinsic viscosity ($\eta_i$) of a dilute polymer solution. This intrinsic viscosity is related to the molecular weight by the following relationship.

$$\eta_i = KM^a$$ (Mark Howink equation)

$\eta_i$ = Intrinsic viscosity.

where

$K$ & $a$ = Constants for a given polymer-solvent combination at a given temperature.

$M$ = Average molecular weight.

Molecular weight constants ($K$ and $a$) for some polymer/solvent systems are given in table III.

Degree of polymerization (DP) is number of repeating unit in the polymer. So increase in DP, molecular weight increases.

CHEMICAL REQUIRED
1. Polystyrene 1%
2. Polystyrene 2%
3. Toluene

### Table II

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Concentration (C) (\text{gm/100ml})</th>
<th>Flow time (t) (sec)</th>
<th>(\eta = \frac{t}{t_0})</th>
<th>(t = \eta \frac{t_0}{\eta_0})</th>
<th>(\eta_{sp} = \eta_{red})</th>
</tr>
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<tr>
<td>I</td>
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<td></td>
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<tr>
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<td>IV</td>
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<td>V</td>
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</table>
PROCEDURE

PREPARATION OF POLYMER SOLUTIONS OF DIFFERENT CONCENTRATIONS.

Polymer solutions different concentrations are prepared by adding particular quantities of solvent (water/org solvent) to the polymer. For example 0.5%, 1.0%, 2.0% and 2.5% are prepared from the given polymer stock solution as in the Table –I.

FOR TIME OF SOLVENT

First flow time of pure solvent is noted by passing the solvent into the viscometer, through the capillary just above the upper mark (M₁) without any air bubbles. When the solvent reaches the mark, the stop clock is switched on and when it reaches the lower mark (M₂) it is switched off. This is the flow time of the pure solvent (t₀).

FLOW TIME OF POLYMER SOLUTION

The flow time of all the polymer solutions are taken as mentioned above. For each polymer solution, wash and rinse the viscometer with the respective solutions.

From the flow times, reduced viscosity ($\eta_{sp} / C$) can be calculated. Graph is plotted between $\eta_{sp} / C$ VS Concentration straight line is obtained with an intercept called intrinsic viscosity ($\eta_i$)
Calculations

Mark-houwink Equation is given by

\[ \eta_i = KM^a \]

\[ \log \eta_i = \log K + a \log M \]

\[ \log M = \frac{\log \eta_i - \log K}{a} \]

\[ M = A \cdot \log \left[ \frac{\log \eta_i - \log K}{a} \right] \]

Where

- \( M \) = molecular weight of the polymer
- \( \eta_i \) = Absolute viscosity of a polymer solution
- \( \eta_0 \) = Absolute viscosity of a pure solvent
- \( t \) = flow time for the polymer solution
- \( t_0 \) = flow time for the solvent
RESULT

The molecular weight of the given polymer = ---------------

INSTRUCTIONS

1. Prepare the polymer solutions of different concentrations
2. Rinse the viscometer with solvent
3. Pipette out 10ml of solvent in the viscometer.
4. Draw the solvent into the capillary arm either by sucking at the open end
   of the capillary arm (or) by applying pressure by gas into the other arm.
5. Allow the solvent to flow through the capillary. When the liquid passes
   the upper graduation mark, start the stop watch. When the lower meniscus
   crosses the lower mark stop the watch.
6. Note the time for the flow of define volume of solvent to how through the
   capillary. Let it be ‘t₀’.
7. Follow the above procedure; find out the flow time of polymer solutions.
   Let it be ‘t’.
8. Repeat this 4 times and it should agree within 0.1sec.
9. Plot ηₛ / C Vs C

SHORT PROCEDURE

Polymer solutions of different concentrations are prepared by adding
particular quantities of solvent to the polymer.

First flow time of pure solvent is noted by passing the solvent into the
viscometer through the capillary tube above the mark with out any air
bubbles.

When the solvent reaches the mark, the stop clock is switched or when it
reaches the lower mark it is switched off. This is the flow time of pure
solvent ‘$t_o$’. Follow the above procedure; find out the polymer solution flow time denoted as ‘t’.

**VIVA VOCE**

1. Polymer is a macromolecule which is obtained by combination of smaller molecules called monomer.
2. The strength of polymer is depend on the molecular weight of the polymer.
3. Polymer classified into mono polymer and copolymer.
4. Degree of polymerization is number of repeating units in the polymer.
5. Mark – Hawink Equation.

\[
\eta_i = KM^a, \\
M = A \log \left[ \frac{\log \eta_i - \log K}{a} \right]
\]
### Some Important Compounds and Its Molecular weights:

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<tr>
<th>Sl.No</th>
<th>Compounds</th>
<th>Molecular weights</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>NH₄Cl</td>
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<td>2</td>
<td>(NH₄)₂HPO₄</td>
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<td>3</td>
<td>NH₄H₂PO₄</td>
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<td>(NH₄)₂SO₄</td>
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<td>5</td>
<td>FeSO₄·7H₂O</td>
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<td>6</td>
<td>FeSO₄·(NH₄)₂SO₄·6H₂O</td>
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PREPARATION OF SOLUTIONS

SOME IMPORTANT INDICATORS AND REAGENTS

INDICATORS;

1. **EBT:**
   Dissolve 0.4 g solid EBT indicator in methanol.

2. **40% Ferric ion indicator;**
   Dissolve 40 g AR ferric ammonium sulphate in distilled water and add few drops of 6 M nitric acid and make upto 100ml.

3. **Starch**
   Prepare a paste of starch by dissolving 1g. of soluble starch with a little water. Pour the paste with constant stirring into 100ml of boiling water and boil for 5 minutes. Cool the solution.

4. **Murexide**
   Grind 0.1g of Murexide with 10 g AR potassium nitrate. Use about 50mg of this mixture for each titration.

5. **Diphenylamine**
   Dissolve 2g of Diphenylamine in 100ml of Conc.H₂SO₄.

6. **Methyl orange**
   1g Methyl orange is dissolved in 100ml ethanol.

7. **Phenolphthalein**
   1g Phenolphthalein is dissolved in 100ml ethanol

REAGENTS

1. **1mg/ml or 1ppm CaCO₃ solution**
   Dissolve 1g pure AR CaCO₃ in few ml of dil.HCl and make upto 1litre.

2. **0.01 M EDTA**
   Dissolve 3.72g disodium salt of EDTA in distilled water and make upto 1 litre.

3. **0.1M AgNO₃**
   Dissolve 17.0 g AR AgNO₃ in chloride free distilled water and make upto 1 litre.

4. **0.1N K₂Cr₂O₇**
   Dissolve 4.9 g Pure AR K₂Cr₂O₇ in distilled water and make upto 1litre.
5. **0.1 N FAS**
   Dissolve 39.2 g of pure FAS in distilled water. Add about 20 ml Conc.H₂SO₄ and make upto 1 litre.

6. **0.1N Sodium thiosulphate**
   Dissolve 25g of AR Sodium thiosulphate crystals in distilled water and make upto 1litre.

7. **10% KI**
   Dissolve 10 g pure AR potassium iodide in 100 ml distilled water.

8. **1M NH₄Cl**
   Dissolve 53.5 g pure AR ammonium chloride in distilled water and make upto 1 litre.

9. **Winkler solution**
   15 g of potassium bromide and 2.78 g of potassium bromate are dissolved in 1 litre distilled water.

10. **0.1N BaCl₂**
    Dissolve 12.2 g of BaCl₂ in 1 litre distilled water.

11. **0.1N Na₂SO₄**
    Dissolve 7.1g of Na₂SO₄ in 1 litre distilled water.

12. **0.1N CuSO₄**
    Dissolve 25g CuSO₄ crystals in 1 litre water.

13. **4N H₂SO₄**
    112 ml Conc.H₂SO₄ + 888ml H₂O and make upto 1litre.

14. **0.1N HCl**
    10 ml Conc .HCl + 990 ml H₂O and make upto 1litre.

15. **6 N HNO₃**
    378 ml Conc. HNO₃ + 622 ml H₂O and make upto 1 litre.

16. **0.1N CH₃COOH**
    6 ml of CH₃COOH is dissolved in 1 litre water.

17. **0.1N NaOH**
    4g NaOH pellets are dissolved in 1 litre water

18. **NH₄OH - NH₄Cl buffer of pH - 10**
    Dissolve 70g of AR NH₄Cl in 570 ml of liquor ammonia and make upto 1litre using distilled water.