



CMS COLLEGE OF ENGINEERING AND TECHNOLOGY

Appachigoundanpathy , Coimbatore -32

ENGINEERING CHEMISTRY LABORATORY MANUAL – I

As per ANNA University syllabus
(For First Semester B.E., / B. Tech. students
common to all branches)



**G.SANTHI
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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.
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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

It's 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 advise 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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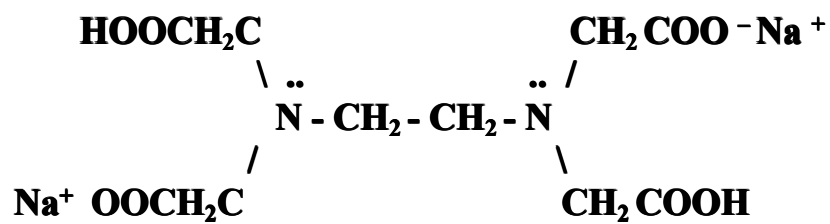
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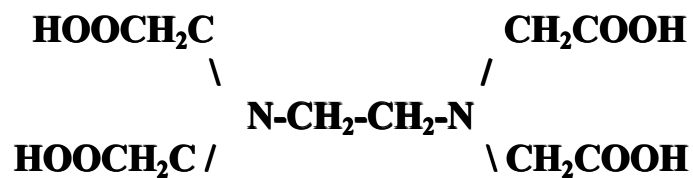
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Internal marks awarded	
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DISODIUM SALT OF EDTA



STRUCTURE OF EDTA



ESTIMATION OF HARDNESS OF WATER SAMPLE - EDTA

METHOD

TITRATION –I

STANDARDISATION OF EDTA

(Std Hard water Vs EDTA)

Sl.No	Volume of Std.Hardwater (ml)	Burette readings		Volume of EDTA solution (ml)	Indicator
		Initial (ml)	Final (ml)		
					EBT

Concordant Value $V_1 =$ _____ ml

CALCULATION:

Volume of Std.Hardwater = 20 ml

Volume of EDTA consumed $V_1 =$ _____ ml (Concordant Value)

1 ml of Std.Hardwater contains 1 mg of CaCO_3

20 ml of Std.Hardwater contains 20 mg of CaCO_3

But

20 ml of Std.Hardwater consumes V_1 ml of EDTA

(ie) V_1 ml of EDTA Solution = 20 mg of CaCO_3

1 ml of EDTA Solution = $\frac{20}{V_1}$ mg of CaCO_3
= mg of CaCO_3

REAGENTS REQUIRED:

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

PROCEDURE:

TITRATION - I

STANDARDISATION OF EDTA

(Std. Hard water Vs EDTA)

The burette 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)

Sl.No	Volume of water sample (ml)	Burette readings		Volume of EDTA solution (ml)	Indicator
		Initial (ml)	Final (ml)		
					EBT

Concordant Value $V_2 =$ _____ ml

CALCULATION:

Volume of water sample = 20 ml

Volume of EDTA consumed $V_2 =$ _____ ml (Titre Value)

20 ml of given water sample consumes V_2 ml of EDTA

(ie) 20 ml of given water sample contains = $\frac{20}{V_1} \times V_2$ mg of CaCO_3

1000 ml of given water sample contains = $\frac{20}{V_1} \times V_2 \times \frac{1000}{20}$ mg of CaCO_3

(ie) = $\frac{V_2}{V_1} \times 1000$ mg of CaCO_3

Total Hardness of given water sample = ----- 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 V_2 ml.

TITRATION -III

ESTIMATION OF PERMANENT HARDNESS

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

Sl.No	Volume of boiled water (ml)	Burette readings		Volume of EDTA solution (ml)	Indicator
		Initial (ml)	Final (ml)		
					EBT

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 = $\frac{20}{V_1} \times V_3$ mg of CaCO_3

1000 ml of given water sample contains = $\frac{20}{V_1} \times V_3 \times \frac{1000}{20}$ mg of CaCO_3

(ie) = $\frac{V_3}{V_1} \times 1000$ mg of CaCO_3

Permanent Hardness of given water sample = ----- ppm

ESTIMATION OF PERMANENT HARDNESS

100ml of given water sample is taken in a clean 250ml beaker and boiled for 10 - 15 minutes. It is then cooled and filtered. The filtrate is collected in a 100ml SMF and made 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 V_3 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

= -----

= ----- ppm

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

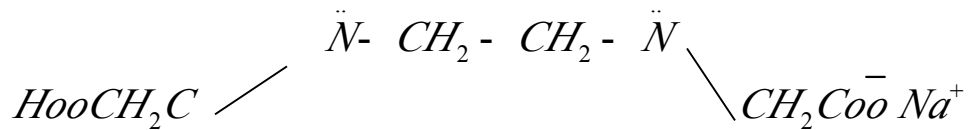
ESTIMATION OF TOTAL HARDNESS

- 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



6. Buffer is a solution used to maintain the p^{H} range of a reaction.

Buffer can be classified into two types namely Acidic & Basic

Example for acidic buffer: $\text{CH}_3\text{COOH} / \text{CH}_3\text{CooNa}$

(Acetic acid/Sodium acetate)

Example for basic buffer: $\text{NH}_4\text{OH} / \text{NH}_4\text{Cl}$

7. Reason for wine red colour



Complex

Hard water

wine red colour

8. Reason for steel blue colour



(Wine Red Colour)

(Steel Blue Colour)

9. Expansion of P^{H} is potential Hydrogen

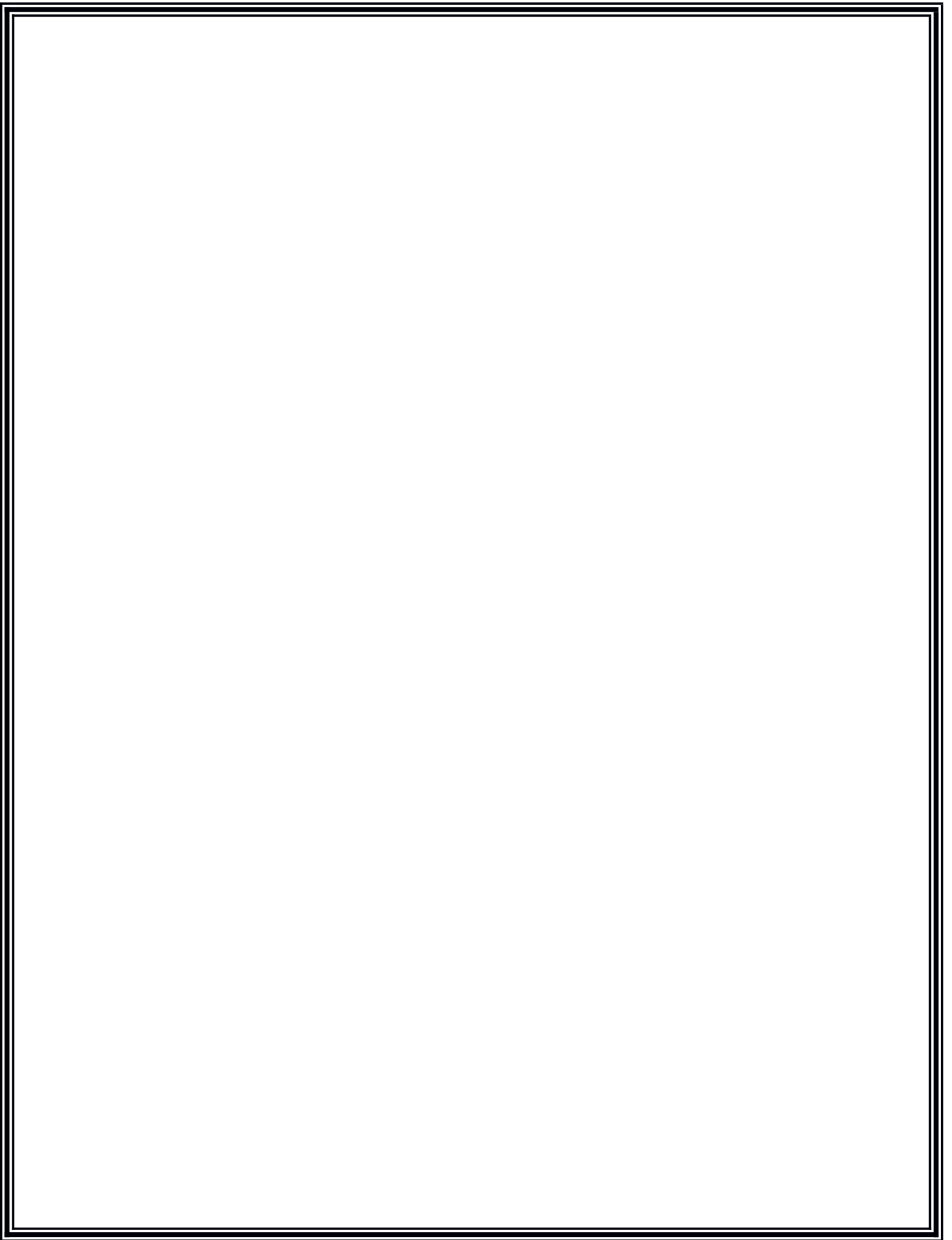
$$\text{P}^{\text{H}} = - \log [\text{H}^+]$$

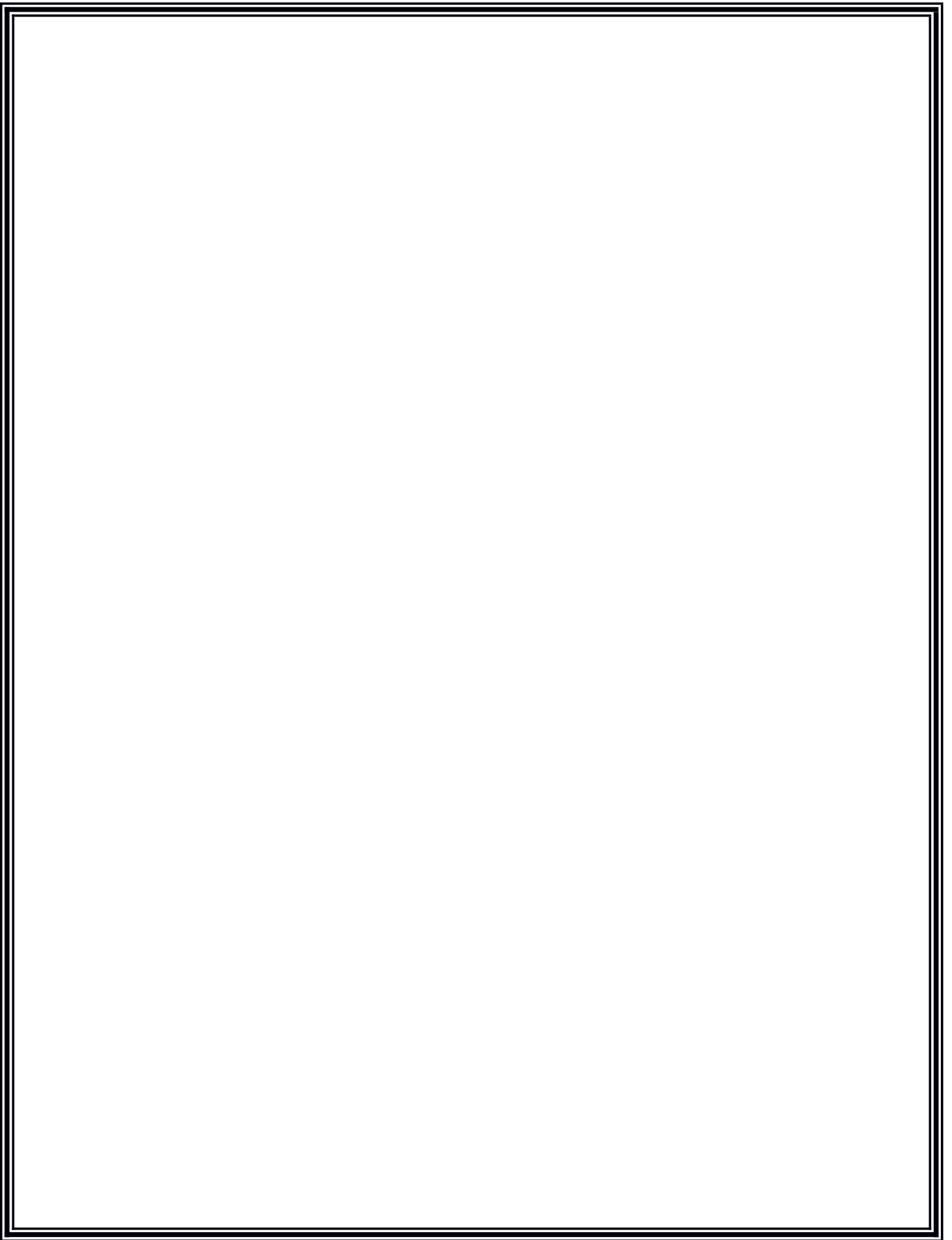
10. P^{H} Scale:

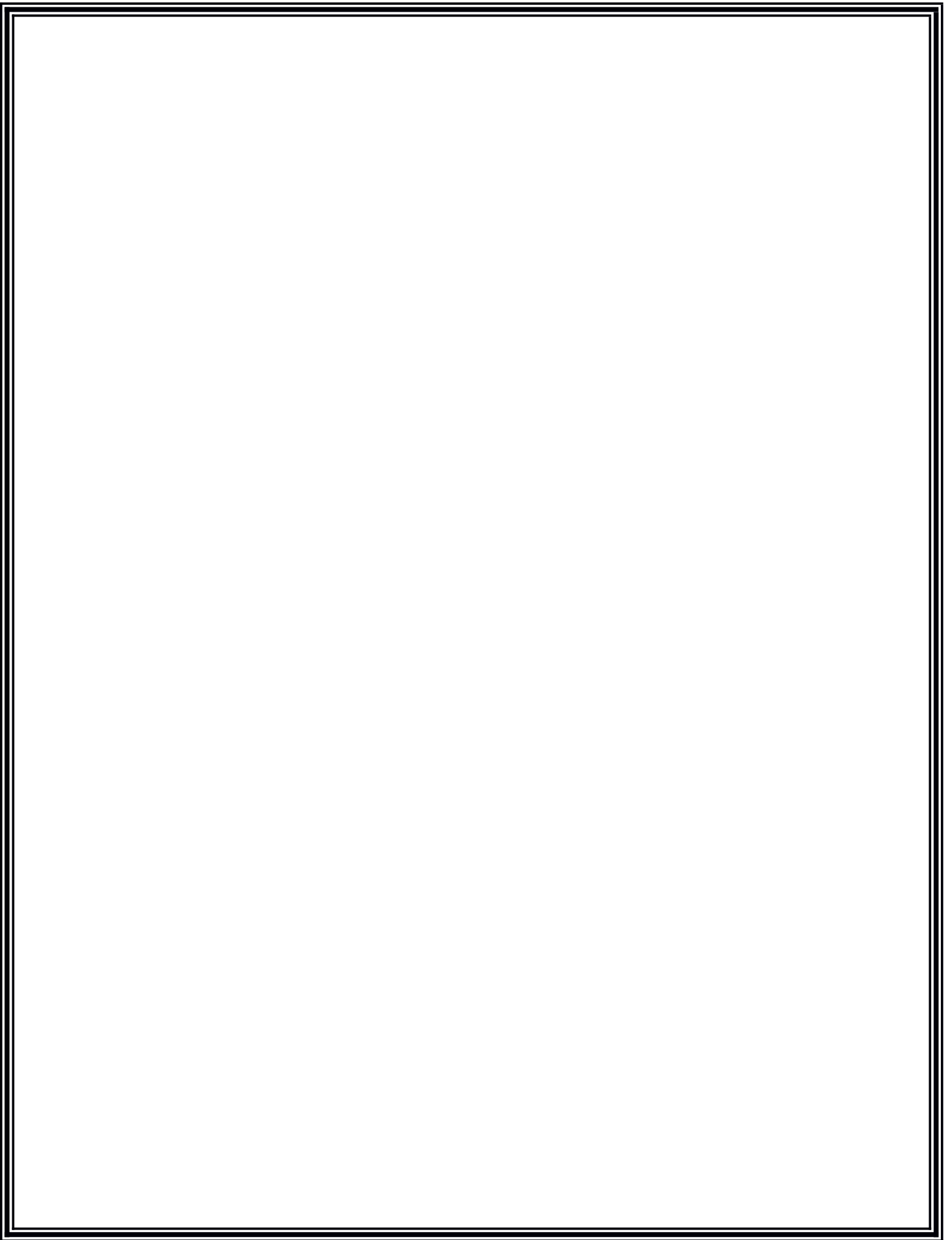
if $\text{p}^{\text{H}} = 7$ the solution is neutral (for pure water)

$\text{p}^{\text{H}} = 0$ to 7 (for acids)

$\text{p}^{\text{H}} = 7$ to 14 (for bases)







ALKALINITY CONDITIONS

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

Sl.No	Result of Titration [P] and [M]	Alkaline causing ions		
		OH ⁻	CO ₃ ²⁻	HCO ₃ ⁻
1	[P] = 0	Nil	Nil	[M]
2	[P] = [M]	[P] or [M]	Nil	Nil
3	$[P] = \frac{1}{2}[M]$	Nil	2[P] or [M]	Nil
4	$[P] > \frac{1}{2}[M]$	2[P] - [M]	2[M] - 2[P]	Nil
5	$[P] < \frac{1}{2}[M]$	Nil	2[P]	[M] - 2[P]

Table – 1

DETERMINATION OF ALKALINITY OF WATER SAMPLE

Expt.No.

Date:

AIM:

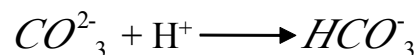
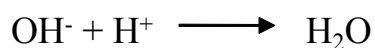
To determine the type and amount of alkalinity present in the given sample of water. A standard NaOH solution of strength ----- N and HCl solutions are provided.

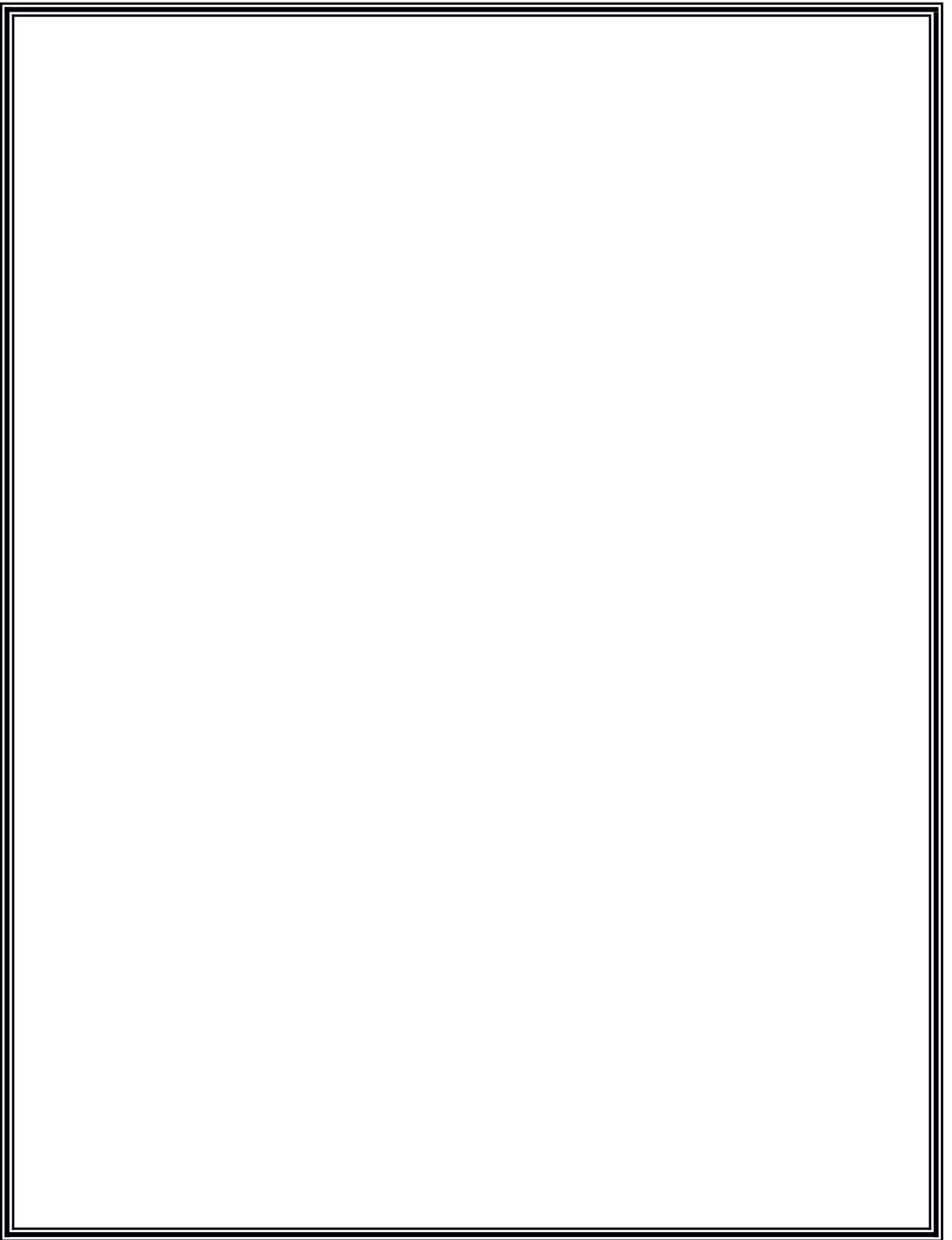
PRINCIPLE:

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:

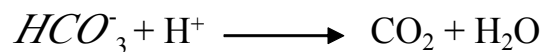
Hydroxide (OH^-) ion alkalinity is completely neutralized and carbonate (CO_3^{2-}) alkalinity is partially neutralized during phenolphthalein end point.





(ii) Methyl Orange End point:

Bicarbonate ion neutralize during methyl orange end point only



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

If, [P] = O; only bicarbonate ion is present

[P] = M; only hydroxide ion is present

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

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

[P] > 1 / 2 M; 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 acid (0.02N), Hydrochloric acid (0.1N), Phenolphthalein (0.5%), Methyl orange (0.5%)

(Std.NaOH Vs HCl)

Sl.No	Volume of NaOH (ml)	Burette readings		Volume of HCl (ml)	Indicator
		Initial (ml)	Final (ml)		
					Phenolphthalein

Concordant Value = _____ ml

CALCULATION:

Volume of NaOH Solution
 Strength of NaOH Solution
 Volume of HCl
 Strength of HCl

$$\begin{aligned}
 V_1 &= 20 \text{ ml} \\
 N_1 &= \text{_____ N (given)} \\
 V_2 &= \text{_____ ml (Concordant Value)} \\
 N_2 &= \frac{V_1 N_1}{V_2}
 \end{aligned}$$

$$20 \times \text{_____}$$

=

Strength of HCl = _____ N

PROCEDURE:

TITRATION - I
STANDARDISATION OF HCl
(STD. NaOH V_s 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)

Sl.No	Volume of Water sample (ml)	Burette readings			Volume of HCl (ml)		Indicator
		Initial (ml)	Final (ml)		P	M	
			P	M			
						I)Phenolphthalein II)Methyl orange	

Concordant Value ; P = _____ ml

M = _____ ml

$\frac{1}{2}M =$ _____ ml

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 1-2drops 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 X -----
= -----ml

Strength of HCl N_1 = _____ N (I calculation)

Volume of water sample V_2 = 20 ml

Strength of water sample N_2 = $\frac{V_1 \cdot N_1}{V_2}$

Strength of Water Sample = _____ N

Amount of CO_3^{2-} ion present in

1 lit of water sample in terms of CaCO_3 equivalent

= Eq. Wt of CaCO_3 x strength of water

sample

= 50 x 1000 ppm x _____ N

Alkalinity due to CO_3^{2-} ions = _____ ppm

HCO_3^- ion alkalinity:

Volume of HCl $V_1 = [M] - 2[P]$

= -----ml

Strength of HCl N_1 = _____ N (I calculation)

Volume of water sample V_2 = 20 ml

Strength of water sample N_2 = $\frac{V_1 \cdot N_1}{V_2}$

Strength of Water Sample = _____ N

Amount of HCO_3^- ion present in

1 lit of water sample in terms of CaCO_3 equivalent

= Eq. Wt of CaCO_3 x strength of water sample

= 50 x 1000 ppm x _____ N

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:

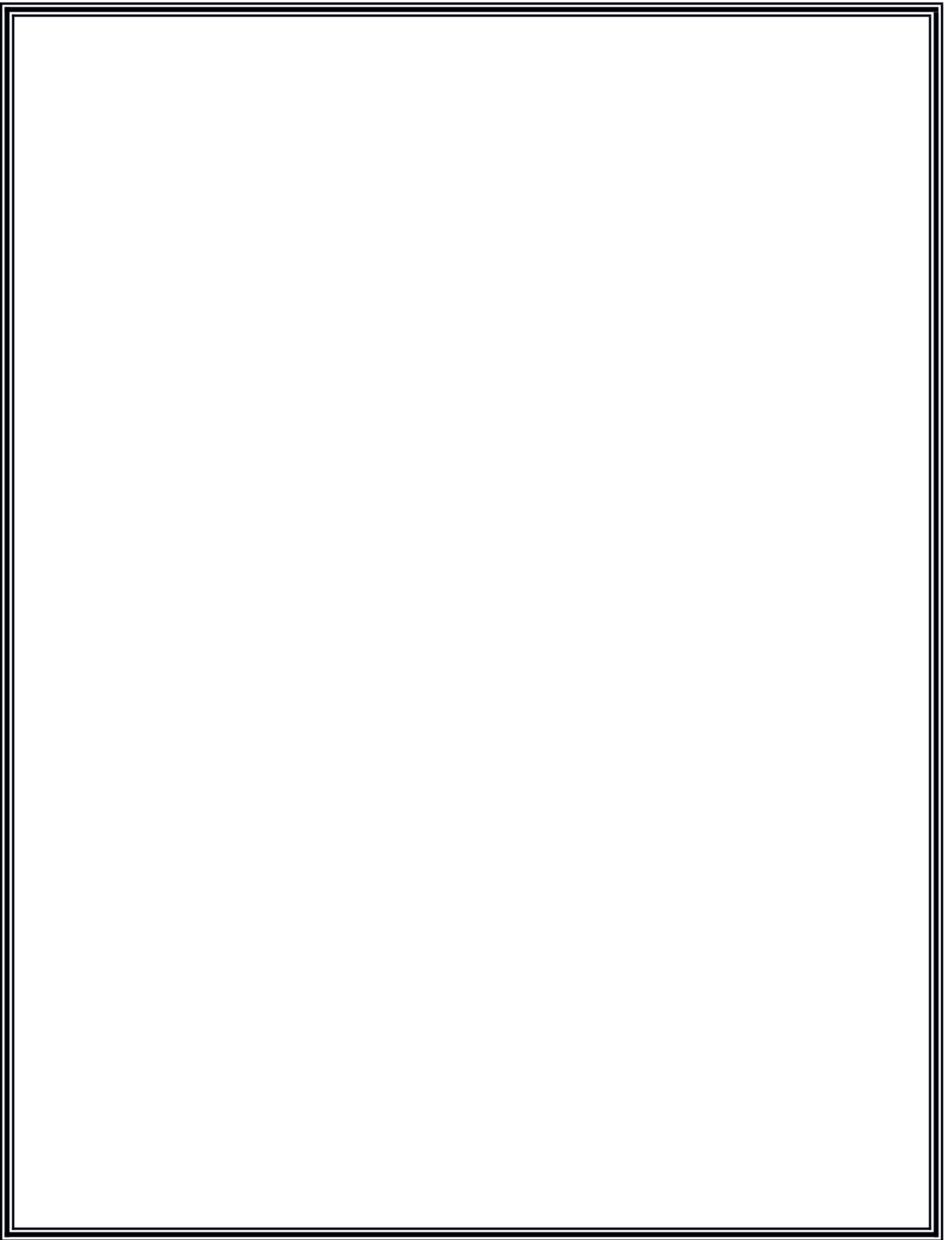
- Fill burette with standardized acid
- Pipette out 20ml of sample water in clean conical flask
- Add one drop of 0.1N thio to remove the free residual chlorine if present.
- Add 2 drops of phenolphthalein indicator
- Titrate the pink solution against acid till colour changes from pink to colourless
- 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.

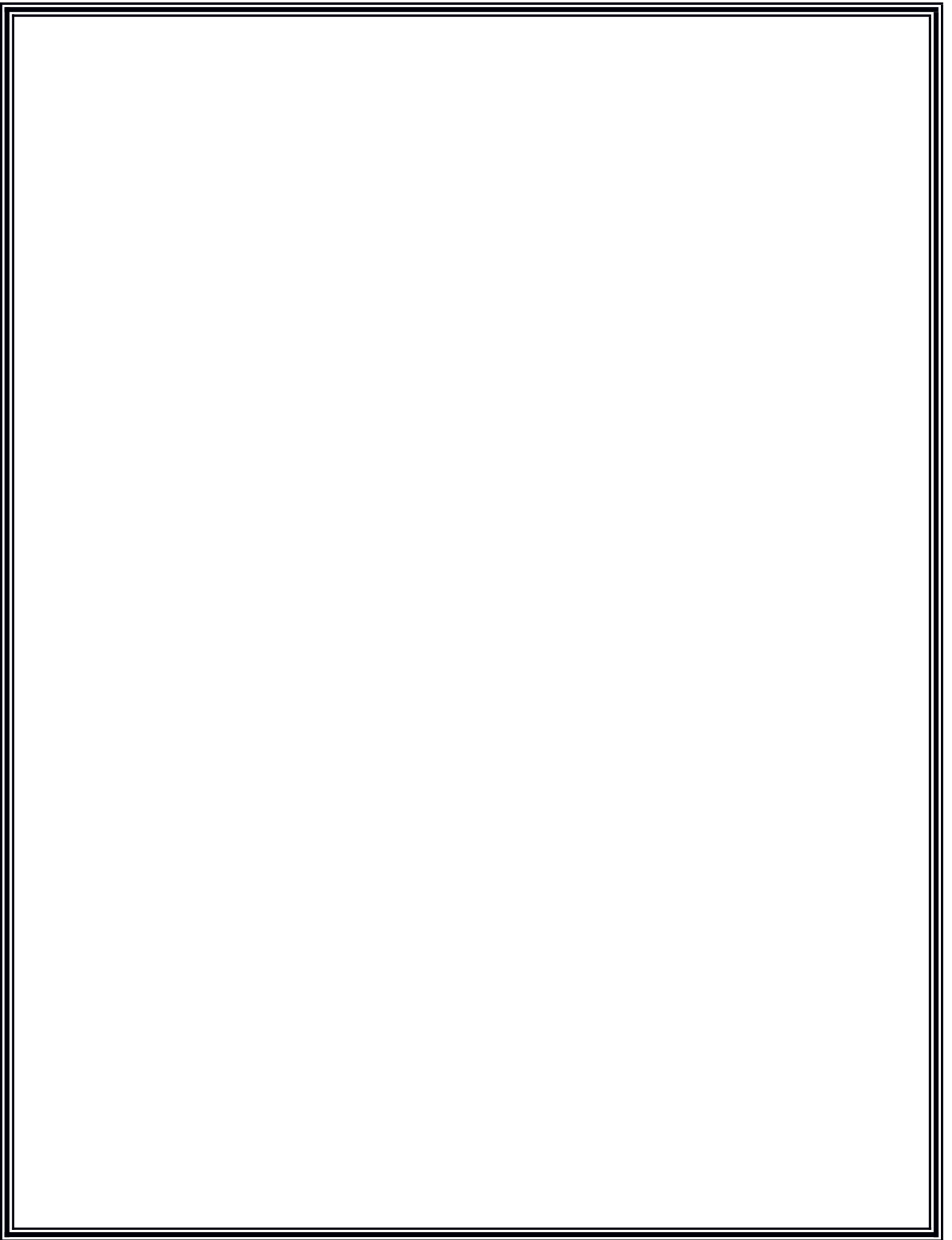
Viva Voce:

- 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 form 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

	Standardisation of HCl Std (NaOH V _s HCl)	Estimation of Alkalinity (Water sample V _s HCl)
Burette solution pipette solution	HCl Std NaOH, 20ml	HCl Water sample, 20ml
Indicator	Phenolphthalein	i) Phenolphthalein ii) Methyl orange
End point	Disappearance of pink colour	i) Disappearance of pink colour. ii) Appearance of orange- pink colour.





ESTIMATION OF DISSOLVED OXYGEN (DO) IN WATER

SAMPLE (WINKLER'S METHOD)

Expt.No

Date:

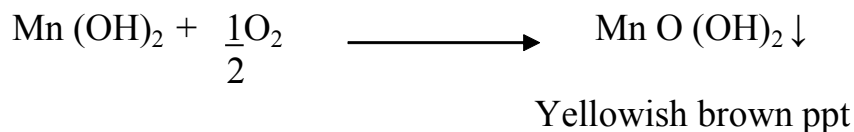
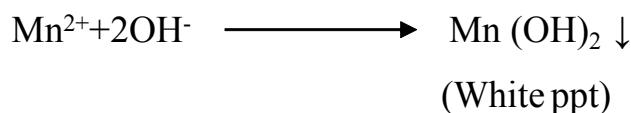
AIM:

To determine the amount of dissolved oxygen (D.O) in tap water by Winkler's method. A standard solution of $K_2Cr_2O_7$ of strength ----- N, sodium thio sulphate are provided.

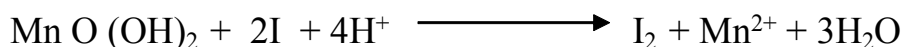
PRINCIPLE:

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 $25^{\circ}C - 35^{\circ}C$.

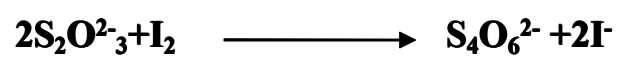
Water sample is collected carefully in a stoppered flask. Manganous sulphate solution in presence of alkali iodide is added to water in an iodine flask. The following reaction occurs.



Dissolved Oxygen in water is utilized for the second reaction. After some time con. H_2SO_4 and KI are added and the reaction is



The known value of this solution is titrated with $Na_2S_2O_3$ solution



STANDARDISATION OF SODIUM THIOSULPHATE

(Std. $K_2Cr_2O_7$ Vs Sodium thiosulphate)

Sl.No	Volume of $K_2Cr_2O_7$ (ml)	Burette readings		Volume of Sodium thiosulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
					Starch

Concordant Value = _____ ml

CALCULATION:

Volume of $K_2Cr_2O_7$ Solution $V_1 = 20$ ml
Strength of $K_2Cr_2O_7$ Solution $N_1 =$ _____ N (given)

Volume of Sodium thiosulphate Solution $V_2 =$ _____ ml (Concordant Value)
Strength of Sodium thiosulphate Solution $N_2 = \frac{V_1 \cdot N_1}{V_2}$

$20 \times$ _____
= _____

Strength of Sodium thiosulphate Solution = _____ N

PROCEDURE:

TITRATION - I

STANDARDISATION OF SODIUM THIO SULPHATE

Burette is filled with sodium thiosulphate. 20ml of standard $K_2Cr_2O_7$ 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)

Sl.No	Volume of Tap water (ml)	Burette readings		Volume of Sodium thiosulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
					Starch

Concordant Value = _____ ml

CALCULATION:

Volume of Sodium thiosulphate Solution $V_1 =$ _____ ml

Strength of Sodium thiosulphate Solution $N_1 =$ _____ N (I calculation)

Volume of Water sample $V_2 =$ 100 ml

Strength of Water sample (or)

Strength of Dissolved oxygen in water $N_2 =$ _____ ?

$$= \frac{V_1 \cdot N_1}{V_2}$$

_____ ml x _____

=

100

= ----- 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.

$$\text{Eq.wt of Oxygen} = 8$$

RESULT

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

Amount of Dissolved oxygen in 1 lit of Tapwater

$$= \text{Eq.Wt of O}_2 \times 1000 \times \text{Normality}$$

$$= 8 \times 1000 \times \text{----- N}$$

$$= \text{----- mgs / lit}$$

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.

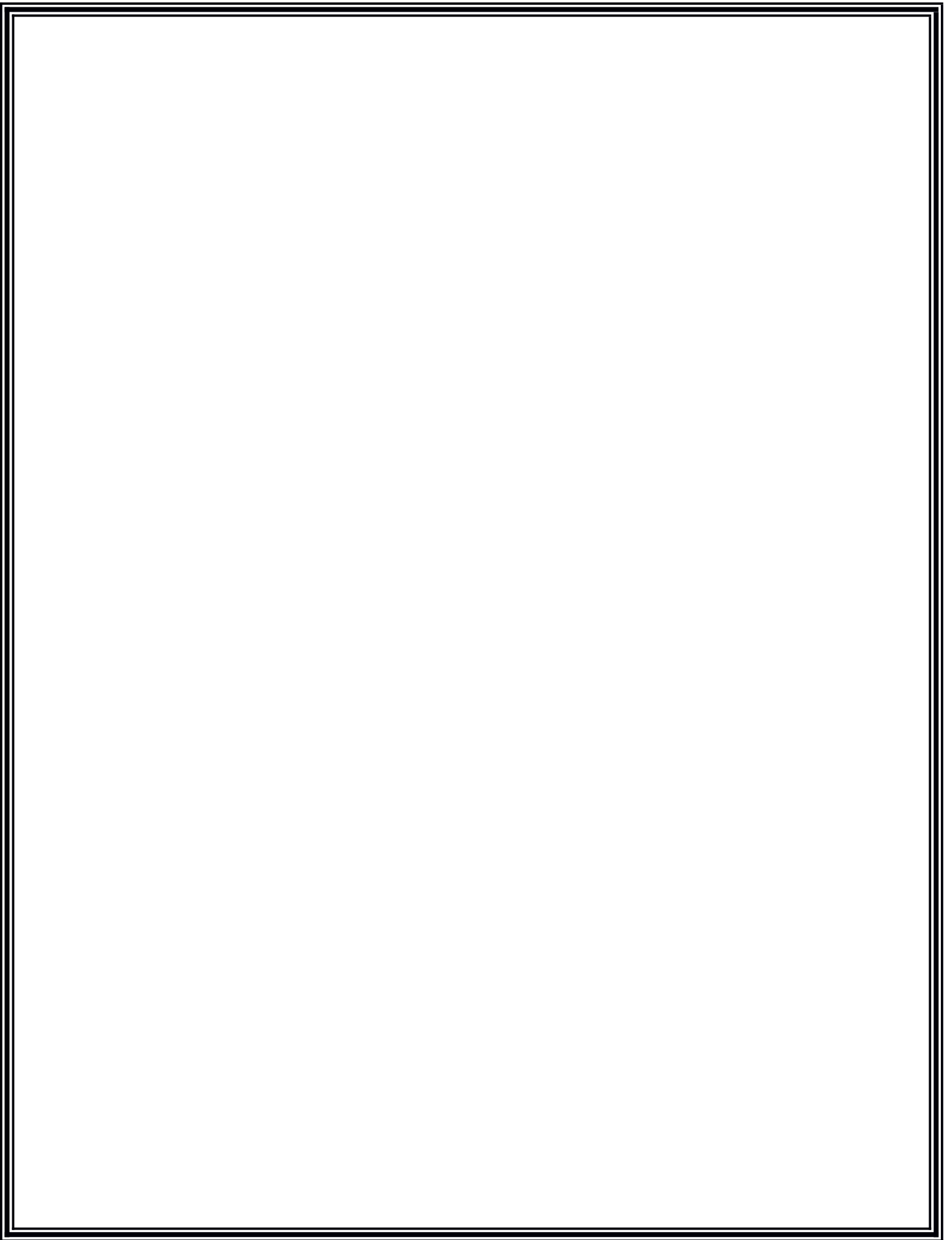
Viva Voce Questions:

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 I_2 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

	Standardisation of Thiosulphate Std $K_2Cr_2O_7$ V_S $Na_2S_2O_3$	Estimate of Dissolved oxygen water sample V_S $Na_2S_2O_3$
Burette Solution	Sodium thiosulphate	Sodium thiosulphate
Pipette Solution	Potassium dichromate	Water Sample (20ml)
Added Solution	5%KI (10ml) + dil H_2SO_4 (10ml)	Alkali Iodide (2ml) + $MnSO_4$ (2ml)
Indicator	Starch	Starch
End Point	Blue colour change to pale green colour	Blue colour change to pale green colour.



DETERMINATION OF CHLORIDE CONTENT OF WATER

SAMPLE - ARGENTOMETRIC METHOD

Expt No.

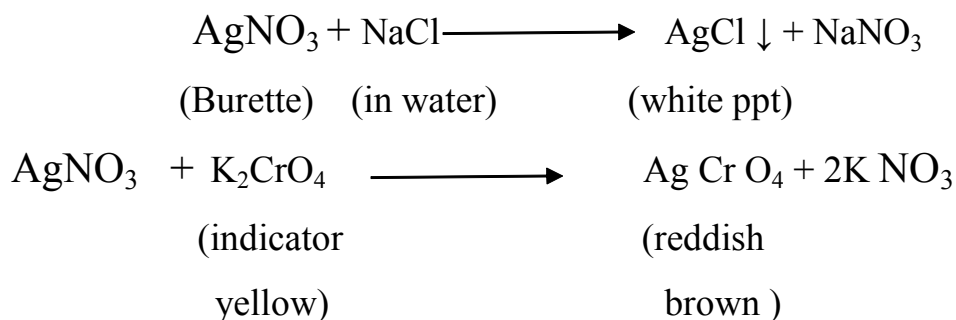
Date:

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,



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)

Sl.No	Volume of NaCl (ml)	Burette readings		Volume of AgNO ₃ (ml)	Indicator
		Initial (ml)	Final (ml)		
					K ₂ CrO ₄

Concordant Value = _____ ml

CALCULATION:

Volume of NaCl Solution
Strength of NaCl Solution

$$V_1 = 20 \text{ ml}$$
$$N_1 = \text{_____ N (given)}$$

Volume of AgNO₃ Solution
(Concordant Value)

$$V_2 = \text{_____ ml}$$

Strength of AgNO₃ Solution
I)

$$N_2 = \text{_____ N (from Titration -$$

$$N_2 = \frac{V_1 \cdot N_1}{V_2}$$
$$= \frac{20 \times \text{_____}}{\text{_____}}$$

Strength of AgNO₃ Solution

$$= \text{_____ N}$$

REAGENTS REQUIRED

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

PROCEDURE

TITRATION - I

STANDARDISATION OF AgNO_3

(AgNO_3 Vs Std. NaCl)

Burette is filled with AgNO_3 solution, pipette out 20ml of std. NaCl solution. To this 1ml of K_2CrO_4 is added as an indicator. It is then titrated against AgNO_3 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)

Sl.No	Volume of Water Sample (ml)	Burette readings		Volume of AgNO ₃ (ml)	Indicator
		Initial (ml)	Final (ml)		
					K ₂ CrO ₄

Concordant Value = _____ ml

CALCULATION:

Volume of Water Sample

$$V_1 = 100 \text{ ml}$$

Strength of Water Sample

$$N_1 = \text{_____ N}$$

Volume of AgNO₃ Solution

$$V_2 = \text{_____ ml (Concordant Value)}$$

Strength of AgNO₃ Solution

$$N_2 = \text{_____ N (from Titration I)}$$

$$N_1 = \frac{V_2 \cdot N_2}{V_1}$$

$$\text{_____} \times \text{_____}$$

$$= \frac{\text{_____}}{\text{_____}}$$

100

Strength of Water Sample

$$= \text{_____ N}$$

Amount of Chloride ion present in

1 lit of water sample

$$= \text{Eq. Wt} \times \text{strength of water sample}$$

$$= 35.45 \times \text{_____}$$

$$= \text{_____ g}$$

TITRATION - II

ESTIMATION OF CHLORIDE ION IN WATER SAMPLE

(Water sample Vs AgNO₃)

Burette is filled with AgNO₃. 100ml of water sample is measured out into a clean conical flask. To this 1ml of K₂CrO₄ is added as an indicator.

It is then titrated against AgNO₃ 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

ESTIMATION OF COPPER IN BRASS BY IODOMETRY

Expt No.

Date:

AIM

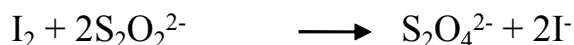
To estimate the amount of Copper present in the brass solution. A standard solution of Pot. dichromate of strength ----- N and sodium thiosulphate as link solution are provided.

PRINCIPLE

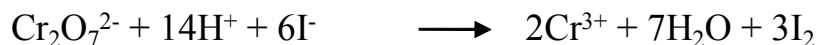
Alloys are the mixture of two or more elements. Brass is an alloy of Copper, iron, Zinc and Tin. It has the percentage of copper 55%.

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 is a brass solution. It is titrated against Na₂S₂O₃ using KI and starch as indicator. The reactions are,



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



(Chromous
ion-Orange)

(Chromic
ion-green)

TITRATION –I

STANDARDISATION OF SODIUM THIOSULPHATE

(Sodium thiosulphate Vs Std.K₂Cr₂O₇)

Sl.No	Volume of K ₂ Cr ₂ O ₇ (ml)	Burette readings		Volume of Sodium thiosulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
					Starch

Concordant Value = _____ ml

CALCULATION:

Volume of K₂Cr₂O₇ Solution V₁ = 20 ml
Strength of K₂Cr₂O₇ Solution N₁ = _____ N (given)

Volume of Sodium thiosulphate Solution V₂ = _____ ml

(Concordant Value)

$$\text{Strength of Sodium thiosulphate Solution } N_2 = \frac{V_1 \cdot N_1}{V_2}$$
$$= \frac{20 \times \underline{\hspace{2cm}}}{\underline{\hspace{2cm}}}$$

Strength of Sodium thiosulphate Solution = _____ 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



Burette is filled with thiosulphate solution. Pipette out 20ml of std. pot.dichromate into a clean conical flask. To this 10ml of dil. H_2SO_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)

Sl.No	Volume of Brass Solution (ml)	Burette readings		Volume of Sodium thiosulphate (ml)	Indicator
		Initial (ml)	Final (ml)		
					Starch

Concordant Value = _____ ml

CALCULATION:

Volume of Sodium thiosulphate Solution $V_1 = 20$ ml

Strength of Sodium thiosulphate Solution $N_1 = \dots\dots\dots$ N (given)

Volume of Brass Solution $V_2 = \dots\dots\dots$ ml (Concordant Value)

Strength of Brass Solution $N_2 = \frac{V_1 \times N_1}{V_2}$

$20 \times \dots\dots\dots$

= _____

Strength of Brass Solution = ----- N

Amount of Copper present in given Brass Solution

= Eq. Wt x strength of Brass Solution

= $63.54 \times \dots\dots\dots$

= _____ 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. H_2SO_4 , 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 in 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 NH_4OH solution is added drop by drop till faint precipitate is obtained. It is then neutralized by adding 2ml of dil. CH_3COOH 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 in 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

Burette Solution	-	Sodium thiosulphate
Pipette Solution	-	Std. Potassium dichromate
Additional Solution	-	10ml of dil.H ₂ SO ₄ +15ml of 10%KI.
Indicator	-	Starch
End point	-	Appearance of green colour.

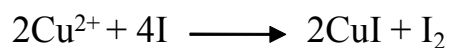
TITRATION – II

Burette Solution	-	Sodium thiosulphate
------------------	---	---------------------

Pipette Solution	-	Brass solution
Additional Solution	-	Few drops NH_4OH +2ml of dil. CH_3CooH +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



3. Sodium thiosulphate also called as thio (or) hypo
4. During the end point Cr^{6+} is converted to Cr^{3+}

Table I

Example for preparation of various concentrated Polymer Solution

Sl.No	Volume of 5% Polymer Solution (Stock solution) (V_1 ml)	Volume of water (ml)	Concentration C (%) (N_2)
I	5	45	$(V_1 N_1 = V_2 N_2)$ $(5 \times 5) \div 50 = 0.5$
II	10	40	$(5 \times 10) \div 50 = 1.0$
III	15	35	$(5 \times 15) \div 50 = 1.5$
IV	20	30	$(5 \times 20) \div 50 = 2.0$
V	25	25	$(5 \times 25) \div 50 = 2.5$

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 (η_i) of a dilute polymer solution. This intrinsic viscosity is related to the molecular weight by the following relationship.

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

$$\eta_i = \text{Intrinsic viscosity.}$$

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

Viscosity data for a Polymer / Solvent

Flow time of the pure Solvent (t_0) = ----- sec

Sl.No	Concentration (C) gm/100ml	Flow time t (sec)	$\frac{\eta}{\eta_0} = \frac{t}{t_0} = \eta_r$	$\frac{t}{t_0} = \eta_{sp}$	$\frac{\eta_{sp}}{C} = \eta_{red}$
I					
II					
III					
IV					
V					

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.

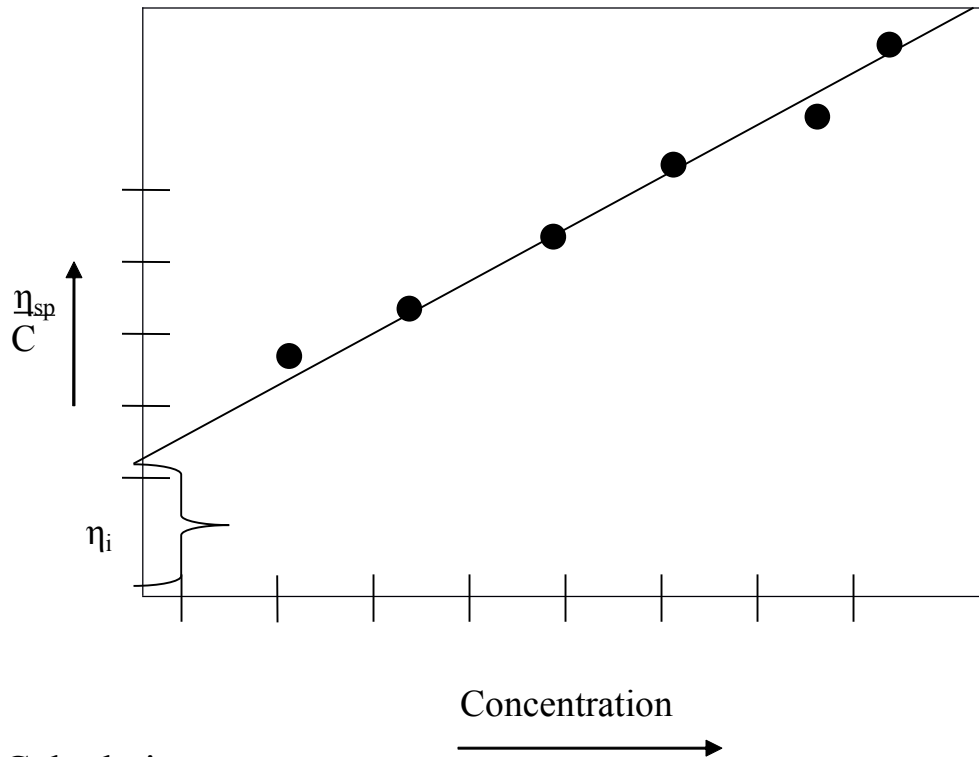
FOR TIME OF SOLVENT

First flow time of pure solvent is noted by passing the solvent into the viscometer, through the capillary just above the upper mark (M_1) without any air bubbles. When the solvent reaches the mark, the stop clock is switched on and when it reaches the lower mark (M_2) it is switched off. This is the flow time of the pure solvent (t_0).

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 (η_{sp} / C) can be calculated. Graph is plotted between η_{sp} / C Vs Concentration straight line is obtained with an intercept called intrinsic viscosity (η_i)



Calculations

Mark- hownik Equation is given by

$$\eta_i = KM^a$$

$$\log \eta_i = \log K + a \log M$$

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

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

Where

M = molecular weight of the polymer

η_i = Absolute viscosity of a polymer solution

η_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 ' t_0 '.
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 η_{sp} / 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 't₀'. 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:

S

Sl.No	Compounds	Molecular weights
1	NH_4Cl	53.49
2	$(\text{NH}_4)_2\text{HPO}_4$	132.06
3	$\text{NH}_4\text{H}_2\text{PO}_4$	115.03
4	$(\text{NH}_4)_2\text{SO}_4$	132.13
5	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	278.01
6	$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	392.13
7	CaCO_3	100
8	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	246.47
9	$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$	223.06
10	KHCO_3	100.12
11	K_2CO_3	138.21
12	$\text{K}_2\text{Cr}_2\text{O}_7$	294.18
13	KI	166
14	K_2SO_4	174.25
15	AgNO_3	169.87
16	KCl	74.55
17	$\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$	136.08
18	$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	286.14
19	NaHCO_3	84.01
20	NaCl	58.42
21	NaOH	40
22	$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$	322.19
23	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$	248.17
24	$\text{C}_6\text{H}_4\text{C}_2\text{O}_4\text{KH}$	204.1
25	HCl	36.45
26	H_2SO_4	98
27	HNO_3	63
28	K_2CrO_4	194.2
29	$\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$	126
30	$\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$	198
31	NH_4SCN	76.12
32	KSCN	97.18

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 P^H - 10**

Dissolve 70g of AR NH₄Cl in 570 ml of liquor ammonia and make upto 1litre using distilled water.

