INDEX

Sr. No.	Experiment	Date Of Performance	Date Of Submission	Marks Obtained Out Of 10	Remark/ Teacher's Sign
1.	Determination of total, permanent and temporary hardness of water by complexometric method.				
2.	Determination of type and extent of alkalinity by Warder's method. (Sample A / B)				
3.	To determine the Normality in normal terms and Strength in gm/lit of HCl solution.				
4.	To estimate the amount of free chlorine present in the given water sample.				
5.	Determination of acid value of oil (Neutralization No.)				
6.	Determination of pH of waste water				
7.	To determine the Normality & Strength of given acid solution by Conductometrically.				
8.	To determine the relative viscosity of given liquid using Oswald's Viscometer				
9.	To determine the physical parameters such as turbidity of a water sample. (V-Lab.)				
10	To find out the unknown concentration of the sample by using Beer-Lambert's Law. (V-Lab.)				

LABORATORY INSTRUCTIONS

-: STUDENTS SHOULD FOLLOW THE INSTRUCTIONS GIVEN BELOW :-

1. Strict silence must be observed during laboratory practice.
2. Keep the working table always neat and clean.
3. Handle the apparatus very carefully.
4. Use aprons at the time of experiment to protect your costly garments.
5. Always keep the reagent bottle at its proper place. Do not change the stoppers of reagent bottles, otherwise it may cause contamination of the reagent.
6. Never return a chemical into the reagent bottle when once taken out, it may cause contamination of reagent.
7. Throw all the rubbish e.g. broken glass test tubes, filter papers, matchsticks etc in dustbins. Do not throw them into sink.
8. Do not waste gas or chemicals. Do not use unnecessary excess of chemicals.
9. Close the water taps and gas taps immediately after use.
10. Strong acids or alkalies should not be thrown directly into the sink. They should diluted before being thrown away.
11. If any accident occurs, report it at once to the teacher concernd.
12. Record the result of all your experiments directly in “laboratory journal” in the prescribed form.

VOLUMETRIC ANALYSIS

INTRODUCTION

- ***It is the process of determining the concentration of an unknown solution by allowing it to react with solution of known concentration (Standard Solution).***
- Measurement required for volumetric analysis is done in terms of volumetric and hence it is known as *'volumetric analysis'/ Titration.*

IMPORTANT TERMS INVOLVED IN TITRATIONS

Standard solution :- The solution of accurately known concentration is called a standard solution.

Titration :- The process of adding standard solution to the solution of unknown concentration until the reaction it just complete is termed as titration.

Indicator :- The completion of the reaction is usually indicated by using a chemical reagent which is known as the indicator.

End Point :- The point at which the reaction it just complete is called *'End Point'* It is generally indicated by change in colour.

Concentration of a solution is usually expressed in following ways.
1. Molarity 2. Normality

Molarity :- It is defined as the numbers of moles of the solute per litre of the solution.

When the molecular weight of the substance is expressed in grams, it is called as gram molecular weight, e. g. 40 gm of solution hydroxide represents 1 gram mol of NaOH.

$$\text{Molarity (M)} = \frac{\text{Number of gm moles of solute}}{\text{Volume of solution in liters}}$$

Equivalent weight :- Equivalent weight of substance is that weight of it which combines with or displaces from chemical combination 1.008 parts by weight of Hydrogen or 8 parts by weight of oxygen or 35.5 parts by weight of chlorine. The equivalent weights of acids and bases are calculated as

$$\text{Equivalent weight (acid)} = \frac{\text{Molecular weight}}{\text{Basicity}}$$

$$\text{Equivalent weight (base)} = \frac{\text{Molecular weight}}{\text{Acidity}}$$

Normality :- The normality (N) of a solution is the number of gram equivalent of the solute present per liter of its solution. A space normal solution will be contained one gram equivalent of the substance per liter of its solution, eg 2N H_2SO_4 means that to gram equivalent of sulphuric acid are contained per liter (2x49=98 grams).

$$\text{Normality (N)} = \frac{\text{Number of gram equivalent Volume of solution}}{\text{Volume of solution}} \qquad \text{Molarity} = \frac{\text{Number of Mole}}{\text{Volume of solution}}$$

It can be also calculated as –

$$\text{Molarity of solution} = \frac{\text{Wt. of substance Dissolved}}{\text{Mol. Wt. of sub. Taken}} \times \frac{1000}{\text{vol. of solution}}$$

$$\text{Normality of solution} = \frac{\text{Wt. of substance Dissolved}}{\text{Eq. Wt. of sub. Taken}} \times \frac{1000}{\text{Vol. of solution}}$$

Law of equivalence : $N_1 \times V_1 = N_2 \times V_2$ OR $M_1V_1 = M_2V_2$
The use of this enables of find the normality / Molarity of unknown solution
The strength of the solution is calculated as
Strength of the solution (gms/liter) = Normality x Equivalent weight

Equivalent weight and molecular weight of reagents:-

Sr. No.	*Name of Compound*	*Formula*	*Molecular Weight*	*Equivalent Weight*
1	Sodium Hydroxide	NaOH	40	40
2	Hydrochloric Acid	HCL	36.5	36.5
3	Sodium Carbonate	Na_2CO_2	106	53
4	Sulphuric Acid	H_2SO_4	98	49
5	Zinc Chloride	$ZaCl_2$	136	68
6	Oxalic Acid	$(COOH)_2$	126	63
7	Potassium Hydroxide	KOH	56	56
8	Ferrous Sulphate	$FeSO_47H_2O$	278	278
9	Ferrous Ammonium Sulphate	$FeSO_4 (NH_4)_2 SO_4 6H_2O$	392	392
10	Sodium Thio-sulphate	$Na_2S_2O_3$-$5H_2O$	248	248
11	Potassium Dichromate	$K_2Cr_2O_7$	294	49
12	Iron	Fe	55.8	55.8
13	Nickel	Ni	58.7	58.7

EQUIVALENT WEIGHT OF OXIDIZING AND REDUCING AGENT

"The equivalent weight of an oxidizing or reducing agent is defined as that weight which directly or indirectly consumes or produces 8 parts by weight of oxygen"
Equivalent weight of some important oxidizing and reducing agent are as follows :

1. $K_2Cr_2O_7 = 294 / 6 = 49$ 2. $KMnO_4 = 31.6$

EXPERIMENT : 0

ZEROTH EXPERIMENT

Aim:- To know your Advanced Chemistry Lab:

The chemistry lab should always be a safe environment for learning and conducting experiments. Following safety protocols is essential, and most of them rely on common sense. Before entering the lab, ensure that you are familiar with the assigned experiment. Always follow written and verbal instructions given by the instructor, and if you have any doubts about the procedure, ask before proceeding.

Once inside the lab, avoid using any equipment, chemicals, or materials unless specifically instructed. Perform only the experiments that have been authorized by the instructor, and stay at your designated station with your assigned lab partners. Do not move to other workstations without permission.

In case of spills, act immediately. If the spill involves a reactive substance such as an acid or base, alert those around you and seek assistance from the teacher. Acid spills should be neutralized with baking soda, while base spills require vinegar. Always be cautious to avoid contamination by keeping chemicals off your hands while washing. Dispose of excess chemicals properly, and never place chemicals directly on the balance or weigh hot objects.

When working with chemicals, never smell substances directly. Instead, use the wafting technique to lift the vapors toward your nose. It is crucial to handle all materials responsibly and keep work areas clean and organized. Always add acid to water while stirring to ensure safety.

Finally, make sure all equipment is cleaned and returned to its proper place at the end of the session.

EXPERIMENT : 1

AIM : Determination of Total, Permanent and Temporary hardness of given water sample by complexometric titration.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand.

CHEMICALS : Standard $ZnCl_2$ solution, EDTA solution, buffer solution of pH 9-10, Erichrome black-T indicator, water sample.

OBSERVATION TABLE OF PART (I) : Standardization Of EDTA

1) Solution in burette = EDTA Solution.
2) Solution in conical flask = 25 ml $ZnCl_2$ solution + 10 ml buffer.
3) Indicator = Erichrome black-T/Solochrome black-T
4) End Point = Wine red to blue.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

CALCULATION : Determination of Molarity of EDTA –

$[ZnCl_2]$ $[EDTA]$

$M_1V_1 = M_2V_2$

EXPERIMENT : 1

AIM : Determination of Total, Permanent and Temporary hardness of given water sample by complexometric titration.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand.

CHEMICALS : Standard $ZnCl_2$ solution, EDTA solution, buffer solution of pH 9-10, Erichrome black-T indicator, water sample.

THEORY : Hardness of water is that property which prevents the lather formation with soap due to the presence of bicarbonate, chloride, sulphate of calcium, magnesium and heavy metals dissolved in water. In short it is the extent of presence of Ca^{2+} & Mg^{2+} ions. Hardness is the soap consuming capacity of water. Soap consists of sodium salts of long chain of fatty acids like palmitic, stearic and oleic acid, Soap react with Ca^{2+} & Mg^{2+} (primary hardness causing agents) to form insoluble scum which has no detergent value.

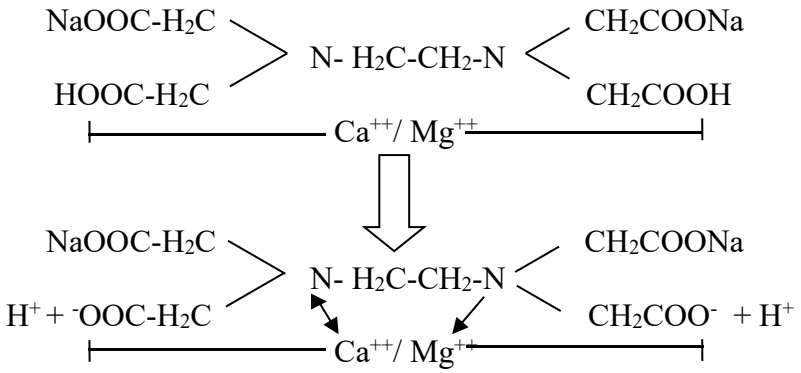
$$2C_{17}H_{35}COONa + Ca^{2+} / Mg^{2+} \longrightarrow (C_{17}H_{35}COO)_2 Ca/Mg + 2Na^+$$

The principle of this titration is that hardness causing ions like Ca^{2+} & Mg^{2+} are forming a complex with EDTA. The amount of EDTA required up to end point is equivalent to hardness present in given water sample. Pure EDTA is sparingly soluble in water. Hence in this experiment di-sodium salt of EDTA is used as a complexing agent which is soluble in water. Here the Erichrome black-T is used as a indicator. EBT is sodium-1-(1-hydroxy 2-napthylazo)-6-nitro-2-naphthol-4-sulphonate(II)

It is blue coloured azo dye. It acts as a metal-ion indicator. It forms unstable wine red complex with metal ions. When this wine red complex is titrated with EDTA solution Ca^{2+} & Mg^{2+} ions from hard water combines with EDTA and indicator is set free at the end point. Hence colour of the solution changes from wine red to blue at end point.

$$M^{++} + EBT \longrightarrow \underset{\text{(wine red unstable comp)}}{M\text{-}EBT} + Na_2EDTA \longrightarrow \underset{\text{(Colourless stable complex)}}{M\text{-}Na_2EDTA} + \underset{\text{(Blue)}}{EBT}$$

The Stability of metal-EDTA complex depends upon the pH of the solution lowering of the pH can decrease the stability of this complex and at high pH indicator also get polymerized. Hence in this titration pH of solution is maintained between pH range 8-10 with the help of alkaline buffer (NH_4Cl and NH_4OH)
EDTA is the complexing agent having the structure:



OBSERVATION TABLE (II) : Determination of Total hardness of water sample.

1) Solution in burette = EDTA
2) Solution in conical flask = 25 ml Water sample + 10 ml buffer solution
3) Indicator = Erichrome black-T
4) End Point = Wine red to blue.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

CALCULATION : Standard Relation : 1ml of 1Molar EDTA = 100mg. of $CaCO_3$

OBSERVATION TABLE (III) : Determination of Permanant hardness.

1) Solution in burette = EDTA
2) Solution in conical flask = 25 ml boiled Water + 10 ml buffer solution
3) Indicator = Erichrome black-T
4) End Point = Wine red to blue.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

The determination of Ca^{2+} and Mg^{2+} hardness separately gives information about exact treatment of water. It helps to calculate the proper quantity of lime and soda. .

Standard Relation : 1ml of 1Molar EDTA = 100mg. of $CaCO_3$

PROCEDURE-I : Standardization Of EDTA

1) All the apparatus are washed with water.
2) Then the burette is rinsed with the EDTA & filled up to zero mark & then clamped on the burette stand.
3) The pipette is rinsed with $ZnCl_2$ solution and 25 ml $ZnCl_2$ solution is pipette out in conical flask.
4) 10 ml buffer solution (NH_4Cl and NH_4OH) is added in conical flask and 2-3 drops of EBT indicator, which gives wine red colour to the solution.
5) This solution is titrated with EDTA Till colour of solution changes from wine red to blue which indicates the end point of titration.
6) The Burette reading is noted.
7) The process is repeated till two consecutive constant reading are obtained.

PROCEDURE-II : Determination Total hardness

1) The burette is rinsed with the EDTA & filled up to zero mark & then clamped on the burette stand.
2) The pipette is rinsed with water sample and 25 ml water sample is pipette out in conical flask.
3) 10 ml buffer solution (NH_4Cl and NH_4OH) and 2-3 drops of EBT indicator is added in conical flask.
4) This solution is titrated with EDTA Till colour of solution changes from wine red to blue which indicates the end point of titration.
5) The Burette reading is noted.
6) The process is repeated till two consecutive constant reading are obtained.

PROCEDURE - (III) Determination Permanant hardness

1) The burette is rinsed with the EDTA & filled up to zero mark & then clamped on the burette stand.
2) Take 100 ml water sample in borosil beaker and boil it for 30 minutes or till volume reduces to half.
3) It is then cool at room temperature & filter it by using filter paper in standard volumetric flask.
4) Then add distilled water and make it up to the mark.
5) Then pipette out 25ml water solution from volumetric flask into conical flask, add ½ test tube buffer solution and 1-2 drops of EBT indicator and titrate with standard EDTA solution.
6) The end point is wine red to blue. Note down end point reading.

CALCULATION : Standard Relation : 1ml of 1Molar EDTA = 100mg. of $CaCO_3$

RESULT :-

1) Total Hardness present in give water sample =
2) Temporary Hardness present in give water sample =
3) Permanant Hardness present in give water sample =

SIGNIFICANCE : This method is useful to find out.

1. Hardness of water which is used in industries like paper, textile, leather etc.
2. Hardness of cooling water & boiler feed water, which may cause scales and sludge formation.
3. Hardness of water which is used for domestic purposes which is obtained from well, rivers, and streams etc.

RESULT :-

1) Total Hardness present in give water sample =
2) Temporary Hardness present in give water sample =
3) Permanant Hardness present in give water sample =

Viva Marks : **Teachers Sign :**
Date :

EXPERIMENT : 2

AIM : To determine type and extent of alkalinity present in the given water sample by warder's method.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand.

CHEMICALS : HCl, Na_2CO_3, water sample A, water sample B, phenolphthalein indicator, and methyl orange indicator.

OBSERVATION TABLE OF PART (I) : Standardization Of HCl

1) Solution in burette = HCl.
2) Solution in conical flask = Na_2CO_3.
3) Indicator = Methyl Orange
4) End Point = Yellow to Orange.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

CALCULATION : Determination of Normality of HCl –

$[Na_2CO_3]$ $[HCl]$

$N_1V_1 = N_2V_2$

EXPERIMENT : 2

AIM : To determine type and extent of alkalinity present in the given water sample by warder's method.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand.

CHEMICALS : HCl, Na_2CO_3, water sample A, water sample B, phenolphthalein indicator, and methyl orange indicator.

THEORY : Alkalinity of natural water may be attributed to the presence of salts of weak acids such as bicarbonate, phosphate, silicates, and borate's which induce buffer capacity and resists the lowering of PH. The alkalinity of water can considered to be mainly due to (a) Hydroxides (b) Carbonates (c) Bicarbonates.
Highly alkaline water may lead to caustic embitterment and also may cause deposition of precipitates and sludge in boiler tubes and pipes.

With respect to the constituents causing alkalinity in natural water, the following situation may rises.

1. Hydroxides only
2. Carbonates only (CO_3^{2-})
3. Bicarbonate only (HCO_3^-)
4. Hydroxides and carbonates (OH^- and CO_3^{2-})
5. Carbonate and Bicarbonates (CO_3^{2-} and HCO_3^-)

NOTE: - The possibility of hydroxides and bicarbonates existing together is ruled out, owing to the fact that they combine with each other forming respective carbonate.

$$OH^- + HCO_3^- \longrightarrow CO_3^{2-} + H_2O$$

The type and extent of alkalinity in water sample may be determined by titrating water sample with standard acid to phenolphthalein end point (P) and then continuing titrating to methyl orange end point (M). The reactions taking place is.

1) $OH^- + H^+ \longrightarrow H_2O$ PH = 7
2) $CO_3^{2-} + H^+ \longrightarrow HCO_3^-$ PH = 8
3) $HCO_3^- + H^+ \longrightarrow H_2CO_3 \longrightarrow H_2O + CO_2$ PH = 4

Volume of acid runs down up to phenolphthalein end point (P) corresponds to the completion of reaction (1) and (2) while volume of acid run down after (P) corresponds to the completion of reaction (3). The amount of acid run down form beginning i.e. (M) corresponds to total alkalinity.

OBSERVATION TABLE (II) : Determination of Alkalinity (A)

1) Solution in burette = HCl
2) Solution in conical flask = Water sample A
3) Indicator = 1] Phenolphthalein (I) 2] Methyl Orange (II)
4) End Point = 1] Pink to colourless (I) 2] Yellow to Orange(II)

Sr. No.	Volume of _____	Burette Reading			Volume of _____used	
		Initial	[P]	[M]	[P]	[M]

CALCULATION :

A] RELATION BETWEEN P & M; P<1/2 M

I) Hence, CO_3^{--} & HCO_3^- Alkalinity present in water sample

i) Alkalinity due to CO_3^{--} :

Volume of HCl equivalent to CO_3^{--} alkalinity = 2p

[Because alkalinity of water is expressed in terms of calcium carbonate equivalent]
.: 1ml of 1N HCl = 50mg of $CaCO_3$
Strength of CO_3^{--} (HCl) = Water sample A

$$N_2V_2 = N_3V_3$$

$$N_3 =$$

Alkalinity due to CO_3^{--} = N_3 x 50 x 1000 = _____ ppm.

Relation between P and M Type of alkalinity		**Extent of alkalinity**		
		OH^-	**CO_3^{2-}**	**HCO_3^{2-}**
P = 0	Only HCO_3^-	Nil	Nil	[M]
P = ½ M	Only CO_3^{2-}	Nil	2P	Nil
P = M	Only OH^-	M	Nil	Nil
P> ½ M	OH^- and CO_3^-	(2P-M)	2(M-P)	Nil
P < ½ M	CO_3^{2-} and HCO_3^-	Nil	2P	(M-2P)
Alkalinity is generally expressed in parts per million in terms of $CaCO_3$				

PROCEDURE PART-I : Standardization Of HCl Solution

1) All the apparatus are first washed with water.
2) The burette is rinsed with the HCl solution.
3) The pipette is rinsed with Na_2CO_3 solution.
4) Take 25ml of 0.1N Na_2CO_3 in conical flask and add 1-2 drops of phenolphthalein indicator, the solution turns pink.
5) Titrate with HCl form burette. At the end point pink colour changes to colorless.
6) The process is repeated till two consecutive constant reading are obtained.

PROCEDURE PART-II
Determination of type and extent of alkalinity (sample A)

1) All the apparatus are first washed with water.
2) The burette is rinsed with the HCl solution.
3) Take 25ml of water sample [A] in conical flask and add two drops of phenolphthalein to it, solution becomes pink.
4) Titrate this solution with HCl form burette. At the end point [P] colour changes from pink to colorless.
5) To this colorless solution add 1-2 drops of methyl orange. Solution becomes Yellow.
6) Titrate this solution with same HCl form the same burette. At the end point [M] Colour changes to pinkish orange.

ii) Alkalinity due to HCO_3^- :
Volume of HCl equivalent to HCO_3^- = [M-2P]

1ml of 1N HCl = 50mg of $CaCO_3$
Strength of HCO_3^- (HCl) = Water sample A

$N_2V_2 = N_4V_4$

N_4 =

Alkalinity due to HCO_3^- = N_4 x 50 x 1000 = _____ ppm.

Total alkalinity of water sample = [Alkalinity due to CO_3^{--} + due to HCO_3^-]

OR

B] RELATION BETWEEN P & M; P>1/2 M
I) Hence, OH^- & CO_3^{--} Alkalinity present in water sample

i) Alkalinity due to OH^- :
Volume of HCl equivalent to OH^- alkalinity = [2P-M]

[Because alkalinity of water is expressed in terms of calcium carbonate equivalent]
.: 1ml of 1N HCl = 50mg of $CaCO_3$
Strength of OH^- (HCl) = Water sample B

$N_2V_2 = N_3V_3$

N_3 =

Alkalinity due to OH^- = N_3 x 50 x 1000 = _____ ppm.

ii) Alkalinity due to CO_3^{--} :
Volume of HCl equivalent to CO_3^{--} = 2[M-P]

1ml of 1N HCl = 50mg of $CaCO_3$
Strength of O CO_3^{--} (HCl) = Water sample B

$N_2V_2 = N_4V_4$

N_4 =

SIGNIFICANCE :-

This method is useful to find out alkalinity of boiler feed water, as alkaline water leads to :

(*a) Caustic embitterment (alkaline corrosion)*

(b) Deposition of precipitate in boiler tubes.

(c) Scales and sludge in pipes

(d) Cause temporary hardness which leads to scale formation in boiler.

Alkalinity due to CO_3^{--} = N_4 x 50 x 1000 = _____ ppm.

Total alkalinity of water sample = [Alkalinity due to OH^- + due to CO_3^{--}]

RESULT : A] 1) The Alkalinity due to CO_3^{--} ion =

2) The Alkalinity due to HCO_3^- ion =

3) Total Alkalinity of water sample =

OR

B] 1) The Alkalinity due to OH^- ion =

2) The Alkalinity due to CO_3^{--} ion =

3) Total Alkalinity of water sample =

RESULT : A] 1) The Alkalinity due to CO_3^{--} ion =

2) The Alkalinity due to HCO_3^- ion =

3) Total Alkalinity of water sample =

OR

B] 1) The Alkalinity due to OH^- ion =

2) The Alkalinity due to CO_3^{--} ion =

3) Total Alkalinity of water sample =

Viva Marks :

Teachers Sign :
Date :

EXPERIMENT: 3

Aim: To determine the Normality in normal terms and Strength in gm/lit of HCl solution.

Apparatus: Burette, pipette, conical flask, etc
Chemicals: 0.1N Oxalic Acid, Na_2CO_3, HCl solution.
Reactions:

1) $HOOC\text{-}COOH + Na_2CO_3 \longrightarrow NaOOC\text{-}COONa + H_2O + CO_2$
2) $2HCl + Na_2CO_3 \longrightarrow 2NaCl + H_2O + CO_2$

1. **Part I Titration of Oxalic Acid V_s Na_2CO_3**

Observations: Part - I

1) Solution in burette = 0.1N Oxalic Acid
2) Solution in conical flask = Na_2CO_3.
3) Indicator = Methyl Orange
4) End Point = Yellow to Orange.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

Calculations: HNO_3 V_s Na_2Co_3

$N_1 \times V_1 = N_2 \times V_2$

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

Normality of Na_2CO_3 (N_2) =

EXPERIMENT: 3

Aim: To determine the Normality in normal terms and Strength in gm/lit of HCl solution.

Apparatus: Burette, pipette, conical flask, etc
Chemicals: 0.1N Oxalic Acid, Na_2CO_3, HCl solution.
Reactions:

1) $HOOC\text{-}COOH + Na_2CO_3 \longrightarrow NaOOC\text{-}COONa + H_2O + CO_2$

2) $2HCl + Na_2CO_3 \longrightarrow 2NaCl + H_2O + CO_2$

Part I Titration of Oxalic Acid V_s Na_2CO_3
Procedure:

1. Wash the burette and pipette with water.
2. Rinse the burette with Oxalic solution and fill up to zero mark with Oxalic Acid.
3. Pipette out the exactly 10ml Na_2CO_3 solution and transfer it into conical flask. Add 1-2 drops of methyl orange indicator and shake well.
4. Titrate this solution against Oxalic Acid solution with drop by drop and constant stirring.
5. The end point of titration will from yellow to orange and record the burette reading in cm^3.
6. Repeat the procedure for 2-4 times and find out C.B.R.
7. Calculate the normality of Na_2CO_3 solution.

Part II Titration of Na_2CO_3 Vs HCl
Procedure:

1. Wash the burette and pipette with water then rinse the burette with HCl solution.
2. Fill the burette with HCl solution.
3. Pipette out exactly 10 ml of Na_2CO_3 solution in conical flask add 1-2 drops of methyl orange indicator and shake well.
4. Titrate this solution against HCl solution.
5. End point of titration is from yellow to orange.
6. Repeat the same procedure for 2-3 times and find out C.B.R.

Observations: Part - II

1) Solution in burette = HCl
2) Solution in conical flask = Na_2CO_3.
3) Indicator = Methyl Orange
4) End Point = Yellow to Orange.

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

Calculation:

$$HCl \quad V_s \qquad Na_2Co_3$$
$$N_1 \times V_1 = N_2 \times V_2$$
$$N_1 = \frac{N_2 X V_2}{V_1}$$

Normality of HCl (N_1) =

Strength of HCl solution = Normality X equivalent weight

Result:

1) Normality of Na_2CO_3 =----------N
2) Normality of HCl =----------N
3) Strength of HCl =----------gm/lit

Result:

1) Normality of Na_2CO_3 =----------N
2) Normality of HCl =----------N
3) Strength of HCl =----------gm/lit

Viva Marks :

Teachers Sign :
Date :

EXPERIMENT : 4

AIM : To estimate the amount of free chlorine present in the given water sample.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand. Burette, cork, test tube.

CHEMICALS : Potassium iodide, Sulphuric acid, Potassium dichromate, sodium bicarbonate, sodium thiosulphate, starch.

OBSERVATION TABLE OF PART (I) : *Standardization Of Hypo Solution*

1) Solution in burette = Hypo (Sodium thio-sulphate)

2) Solution in conical flask = 25 ml $K_2Cr_2O_7$ + 5ml H_2SO_4 + 5ml KI

Cover it for 2 minutes

Brown colour

Hypo

Pale Yellow

Starch

Deep blue

Hypo

Greenish blue

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

EXPERIMENT : 4

AIM :To estimate the amount of free chlorine present in the given water sample.

APPARATUS : Burette, Pipette, Conical Flask, Burette stand, Pipette stand. Burette, cork, test tube.

CHEMICALS : Potassium iodide, Sulphuric acid, Potassium dichromate, sodium bicarbonate, sodium thiosulphate, starch.

THEORY : The process of destroying the pathogenic bacteria and microorganisms of water is known as ***sterilization*** or ***disinfection.*** The chemicals used for this process are called ***disinfectants.*** The various methods used for disinfection of water includes boiling, addition of bleaching powder, chlorine, U.V. radiations etc. however chlorine of water is slightly used for disinfecting water supply since chlorine is powerful oxidizing agent and is cheaply available.
When chlorine is added to water, it forms ***hypochlorous acid,*** which have immediate disastrous effect on microorganisms

$$\underset{\text{Bleaching powder}}{CaOCl_2 + H_2O} \longrightarrow Ca(OH)_2 + Cl_2$$

$$Cl_2 + H_2O \longrightarrow \underset{\text{Hypochlorous acid}}{HOCl} + HCl$$

$$HOCl \longrightarrow \underset{\text{Nascent Oxygen}}{[O]} + HCl$$

$$[O] + \text{Germs} \longrightarrow \text{Killing of Germs}$$

However excess of free chlorine in drinking water is undesirable. An over dose of chlorine chlorine impart an unpleasant taste to eater and is also injurious to health. Hence free chlorine determination is necessary in those of water that have been treated with chlorine.
The principal involved in estimation of free chlorine is that the measured quantity of water is treated with excess amount of potassium iodide solution. The free chlorine present in water brings about the oxidization of potassium iodide and generates equivalent iodine. This liberated iodine in estimated by titrating it against standard hypo solution.

CALCULATION OF PART I –

Normality Of Hypo = $N_1 \times V_1 = N_2 \times V_2$

OBSERVATION TABLE (II) : For free chlorine

1) Solution in burette = Hypo (Sodium thio-sulphate)
2) Solution in conical flask = 25 ml Cu^{++}solution
3) Indicator = Starch
4) End Point = Deep blue to colourless

Sr. No.	Volume of _____	Burette Reading		Volume of _____used
		Initial	Final	

CALCULATION OF PART –II

Normality of Water sample = $N_2V_2 = N_3V_3$

Strength Of Free Chlorine = N_3 x Eq. Wt. Of Chlorine

=

= _____gm/l

Strength Of Free Chlorine in water sample =

RESULT :

1. Normality of Hypo Solution = _____N
2. Amount of free Chlorine = _____gm / lit

PROCEDURE : PART (I)

1. All the apparatus are washed and rinsed with respected solutions.
2. The burette is rinsed with the $Na_2S_2O_3$ solution and burette filled with the same solution.
3. The burette is clamped on the burette stand.
4. The air gap is removed from the nozzle and the level of the solution is adjusted up to zero mark.
5. The pipette is rinsed with $K_2Cr_2O_7$ solution
6. 25 ml of $K_2Cr_2O_7$ solution is pipette out and then transferred into the conical flask. Then 5ml of dilute sulphuric acid and 5ml KI is added and the flask is covered with watch glass for 2minutes till the colour of the solution turns dark brown.
7. It is titrated with hypo solution till the colour changes from dark brown to yellow
8. 4-5 drops of starch indicator is added which turns the solution deep blue.
9. The deep blue solution is again titrated with the hypo solution till colour changes to light blue (end point).
10. The Burette reading is noted at the procedure is repeated till two consecutive constant reading are abtained.
11. The process is repeated till two consecutive constant reading are obtained.

PROCEDURE : PART (II)

1. The burette is filled with hypo solution up to zero mark.
2. 25 ml water sample is pipette out in the conical flask and $^1/_2$ test tube Kl solution is added.
3. The flask is covered with watch glass for 2 to 3 minutes.
4. 2 to 3 drops of starch indicator is added to the flask.
5. This solution is titrated with hypo solution with deep solution changes to colourless at the end point.
6. The burette reading is noted.
7. The procedure is repeated to get two consecutive constant reading.

RESULT :

1. Normality of Hypo Solution = _____N

2. Amount of free Chlorine = _____gm / lit

Viva Marks : **Teachers Sign :**
Date :

EXPERIMENT: 5

Aim : Determination of acid value of oil (Neutralization No.)

APPARATUS: Water bath, burette, pipette, conical flask, burette stand, pipette stand triploid stand.

CHEMICAL: 0.1 N KOH, HCL, Neutral ethyl alcohol, phenolphthalein indicator.

OBSERVATION (I) : <u>Standardization of KOH</u>

1) solution in burette = KOH
2) solution in conical flask = HCL
3) indicator = phenolphthalein
4) End point = colorless to pink

Sr .No.	Volume of _____	Burette reading		Volume of _____ used
		Initial	Final	

CALCULATION: **Determination of Normality of NaOH**

[HCl] = [KOH]

N_1V_1 = N_2V_2

x 25 = N_2 x

OBSERVATION TABLE (II) :determination of acid value

1) Solution in burette = KOH
2) SOLUTION IN Conical flask = oil + solvent
3) Indicator = Phenolphthalein
4) End pint = Coloureless to pink

Sr no	Volume of	Burette reading	Volume of used

EXPERIMENT: 5

Aim: Determination of acid value of oil (Neutralization No.)

APPARATUS: Water bath, burette, pipette, conical flask, burette stand, pipette stand triploid stand.

CHEMICAL: 0.1 N KOH, HCL, Neutral ethyl alcohol, phenolphthalein indicator.

THEROY: The acid value of lubricating oil is defined as the number of mg of KOH required to neutralize free acid present in one gm of the oil. Presence acid in lubricating oil is rare. Free organic acids of acidic bodies always found in lubricating oils. Acid contain should be determined not because it gives any direct evidence of corrosion hazards but to sound to warning that a corrosion test might reveling in good lubricating oil, the acid value should be very low (<0.10) increases in acid value should be taken as an indicator or oxidation of the oil which may lead to gum & sludge formation bodies corrosion. High acid value causes the detoration of oil due to which due to oxidation which form sludge &gum, this spoil the lubrication process. The acid value of fatty oil may vary from 0.2 to50.

$$C_{17}H_{35}COOH + KOH \longrightarrow C_{17}H_{35}COOK + H_2O$$

Formula for determination of acid vlue

$$\text{Acid value} = \frac{\text{No of ml of N/10 KOH used}}{\textit{Wt of oil taken in [gm]}} \times 5.6$$

PROSEDURE PART-1: standardization of KOH solution

1) Burette is washed with water &rinsed with KOH
2) Burette is filled with KOH solution up to the zero mark.(if there is any air bubble in the jet of the burette, it is removed. The level of KOH solution is adjusted to zero mark.)
3) The pipette is washed with water then rinse with HCL.
4) 25 ml of HCL IS PIPETTE out in conical flask & 2-3 drops of phenolphthalein indicator are added in the conical flask.
5) The solution in the conical flask is titrated with given KOH TAKEN IN BURETTE TILL THE permanent light pink colour is obtained. The burette reading is recorded.
6) The above procedure is repeated to gate constant reading.

PROCEDURE PART – II

1. 5 gm of oil is weight out accurately in 250 mi conical flask. 25 ml of neutral alcohol is added.
2. Then it is heated on water bath about 20 min.
3. Burette is rinsed with KOH &filled with KOH solution up to the zero mark. The flask is cooled the room temperature. 2-3 drops of phenolphthalein light pink color appears at end point.
4. The burette reading is noted.

OBSERVATIONS

1) Weight of empty weighing bottle. = $(W_1)g$
2) Weight of empty bottle + 5 gm of oil = $(W_2)g$
3) After transferring of oil &wt of bottle = $(W_3)g$
4) Actual wt of oil taken = (W_2-W_3)

CLCULATION : DETERMINATION OF NORMALITY OF

$$\text{Acid value} = \frac{\text{No. of ml of n/KOH used}}{\text{Wt of oil taken (gm)}} \times 5.6$$

[volume of 0.1n KOH = NORMALITY OF KOH x vol of KOH = 0.1 X VML}

volume of 0.1n KOH = mL

Acid value = x5.6

RESULT:- Acid value of given lubricating oil is found to be= _____.

RESULT:- Acid value of given lubricating oil is found to be= _____.

SIGNIFICANCE:

- Increases in acid value should be taken as an indicator of oxidation of the oil.
- It leads to formation of gm & sludge.
- It leads to corrosion.

CONCLUSION:-

_____

_____

_____

Viva Marks **Teachers Sign:**

Date:

EXPERIMENT : 6

AIM : To determine the pH of a given water samples.

Apparatus: pH meter, Beaker, Wash bottle, Tissue Paper, etc.

Chemicals: Solution of pH 4.0 (Potassium Hydrogen Pthalate 0.05M), Solution of pH 7.0, Solution of pH 9.0 (Borax 0.05M), Distilled water, Saturated KCl Solution.

OBSERVATIONS

Sr. No.	Samples taken	pH measured
1	Water Sample A	
2	Water Sample B	
3	Water Sample C	
4	Water Sample D	

Result: pH of given water samples is found to be –

Sample A-

Sample B-

Sample C-

Sample D-

EXPERIMENT : 6

AIM : To determine the pH of a given water samples.

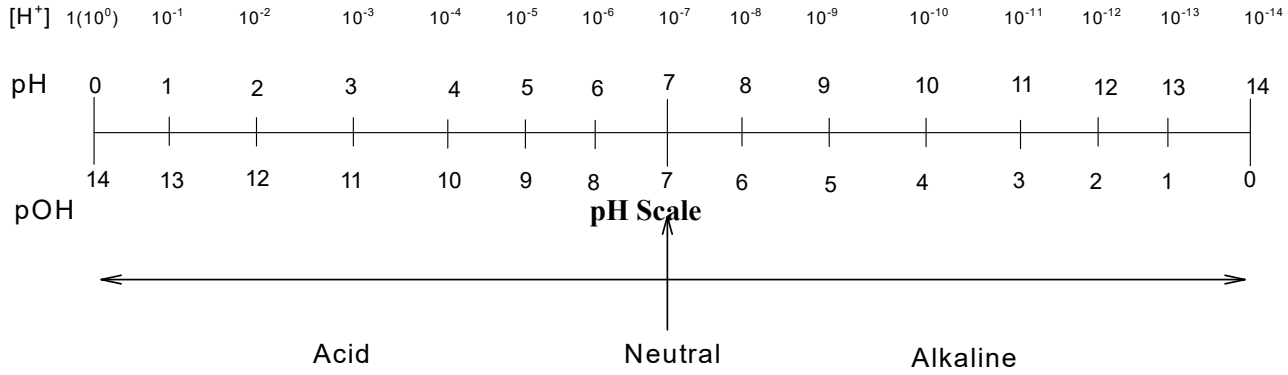
Apparatus: pH meter, Beaker, Wash bottle, Tissue Paper, etc.

Chemicals: Solution of pH 4.0 (Potassium Hydrogen Pthalate 0.05M), Solution of pH 7.0, Solution of pH 9.0 (Borax 0.05M), Distilled water, Saturated KCl Solution.

Theory: For many purpose, especially with small concentrations, it is cumbersome to express concentrations of hydrogen and hydroxyl ions in terms of moles per liter (mol/l). A very convenient method was proposed by S. P. L. Sørensen (1909). He introduced the hydrogen ion exponent pH defined by the relationships-

$$pH = \log_{10}\left(\frac{1}{\left[H^+\right]}\right) = -\log_{10}\left[H^+\right] \text{ or } \left[H^+\right] = 10^{-pH}$$

The quantity pH is thus the logarithmic (to the base 10) of the reciprocal of the hydrogen ion concentration or the logarithm of the hydrogen ion concentration with negative sign. It is used for the determination of the degree of acidity and/or alkalinity of a solution and is expressed by the pH scale which is a series of numbers between 0 to 14.



PROCEDURE OF PART

1. Switch on the instrument and allow it to warm up for some time.
2. Prepare the buffer solutions of 4.0pH, 7.0pH and 9.0pH by dissolving the buffer tablets in specified volume of distilled water. These are necessary for calibration of the instrument.
3. If the instrument is equipped with a manual temperature control, set the control to the measured temperature of the solutions.
4. Insert the electrode assembly into the standard buffer solution (say 7.0pH) taken in a clean and dry beaker.
5. Carefully adjust the "Set Buffer" control until the meter reading display the known pH (7.0pH) of the selected buffer solution.

Result: pH of given water samples is found to be –

Sample A-

Sample B-

Sample C-

Sample D-

Significance:

1. Degree of acidity and/or alkalinity can be determined using pH meter.
2. pH meter can be used for the determination of the end-points in acidimetric and in precipitation titrations.

6. Withdraw the electrode assembly, rinse the electrode with the distilled water and insert it into a beaker containing second buffer solution (say 4.0pH). If the meter reading does not agree exactly with the known pH of the second buffer solution (i.e. 4.0pH), adjust the "Slope" control to achieve the same.
7. Repeat steps from (4) to (6) to ensure the satisfactory calibration using the third buffer solution (i.e. 9.0pH).
8. Withdraw the electrode, rinse with the distilled water and introduced into the "test" (Given water samples) solution taken in another small and clean beaker. Read off pH of the solution, after setting the selector switch to the expected pH range.
9. Remove the electrode assembly out, rinse in distilled water and place it in distilled water taken in another clean beaker. Put back the selector switch to "zero position". Repeat the same procedure for every subsequent pH determinations.

Result: pH of given water samples is found to be –

Sample A-

Sample B-

Sample C-

Sample D-

Significance:

1. Degree of acidity and/or alkalinity can be determined using pH meter.
2. pH meter can be used for the determination of the end-points in acidimetric and in precipitation titrations.

Viva Marks : **Teachers Sign :**
Date :

EXPERIMENT: 7

Aim: To determine the Normality & Strength of given acid solution by Conductometrically.

Apparatus: Conductometer, Beaker, Conductivity cell, Burette, pipette, etc
Chemical: Given acid solution, 0.1N NaOH solution, Distilled water.

Observations:

1. Solution in burette: 0.1N NaOH solution
2. Solution in beaker: 10 ml given acid sample + 40 ml H_2O
3. Temperature of the solution: 30^0C
4. Specific Conductance of KCl
   I) N/10 KCl - 11.99 mM
   II) N/50 KCl - 2.659 Mm

Observation Table:

Sr.No	Volume of 0.1 N NaOH solution in ml	Conductance in mS
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		

Calculation:

Acid = NaOH

$N_1V_1 = N_2V_2$

$N_1 = \frac{N2 \times V2}{V1}$

Normality of acid (N_1) = ----------N

Strength of acid = Normality X Eq. Weight

= (gm/lit)

Cell Constant = $\frac{\text{Specific Conductance}}{\text{Obeseved conductance}}$

Result:

1. Normality of Acid solution = ----------N
2. Strength of acid solution = ----------gms/lit.

EXPERIMENT: 7

Aim: To determine the Normality & Strength of given acid solution by Conductometrically.

Apparatus: Conductometer, Beaker, Conductivity cell, Burette, pipette, etc
Chemical: Given acid solution, 0.1N NaOH solution, Distilled water.

Procedure:

1. Switch on the instrument for 30 min. before starting the experiment.
2. Wash the conductivity cell with distilled water and dry it using filter paper.
3. Conductometer is standardized with KCl solution.
4. Take 10 ml acid in 100 ml beaker and add 40 ml of distilled water.
5. Immerse the Conductivity cell in the solution and measure the Conductance.
6. Add 0.1 N NaOH from burette in this solution cell 1 ml by 1 ml and stir the solution, measure the Conductance
7. Measure the Conductance at each addition of NaOH solution.
8. Plot the graph of Conductance Vs Volume of 0.1 N NaOH added in ml.
9. From the graph determine the equivalence point and calculate the Normality & Strength of the given acid solution.

Result:

3. Normality of Acid solution = ----------N
4. Strength of acid solution = ----------gms/lit.

Viva Marks : **Teachers Sign :**
Date :

EXPERIMENT: 8

Aim: To determine the relative viscosity of given liquid using Oswald's Viscometer

Apparatus: Oswald's viscometer, beaker, stop watch.
Chemicals: Distilled water, acetone and sample liquid (Aniline).
Given:
Viscosity of water: 1.02×10^{-2} poise.

Density of water d_w : $1 gm/cm^3$

Density of Aniline d_l : $1.021 gm/cm^3$

Diagram:

Observation Table:

Sr. No.	Liquid	Density gm / cm^3	Time		Mean t (s)
			t_1(s)	t_2(s)	
1	Water	1.0			t_w =
2	Liquid				t_l =

Calculations:

$$\frac{\eta_l}{\eta_w} = \frac{d_l}{d_w} \times \frac{t_l}{t_w}$$

$$\eta_l = \frac{d_l \times t_l \times \eta_w}{d_w \times t_w}$$

η_l = ---------- poise

Result: The relative viscosity of given aniline =----------poise

EXPERIMENT: 8

Aim: To determine the relative viscosity of given liquid using Oswald's Viscometer

Apparatus: Oswald's viscometer, beaker, stop watch.
Chemicals: Distilled water, acetone and sample liquid (Aniline).
Theory:

When liquid flows through narrow tube, the layer in contact with glass remains stationary and the layers next to it are flowing in the direction of flow. The central layer flows more rapidly. Thus the layers of the liquid are moving with different velocities due to layers. So such layers exerts a drag pressure on another layer due to which there will be restriction. Now the precipitate liquid whatever it may be tries to restrict the flow. Viscosity is the force in dynes required to maintain unit difference in velocities of two adjacent layers.

Viscosity can be determined using the formula.

$$\frac{\eta_l}{\eta_w} = \frac{d_l}{d_w} \times \frac{t_l}{t_w}$$

Where,

η_l , η_w - Viscosities of liquid and water
d_l , d_w - Densities of liquid and water
t_l , t_w - Time required

Procedure:

1. Wash the viscometer with water and then with acetone.
2. Dry viscometer.
3. Take distilled water in wide bulb.
4. Suck it up to mark A.
5. Allow it to flow from A to A'
6. Note down the time required.
7. Repeat the same procedure using aniline instead of distilled water.
8. Note down the time required.
9. Repeat the same procedure to get accurate reading.

Result: The relative viscosity of given aniline =----------poise

Viva Marks : **Teachers Sign :**
Date :