Pollution Control and Safety Management

Laboratory Manual

Chemical Engineering Department

List of Experiments

Experiment Nos.	List of Experiments
--	Introduction to Pollution Control and Safety Management , Briefing about list of experiments with objectives, Assessment policy for experiments
1	Standardization of 0.01M EDTA solution by using 0.01N $CaCO_3$.
2	To determine the hardness of water.
3	To determine the calcium hardness of water.
4	To determine acidity of a given water sample.
5	To determine the alkalinity of a water sample.
6	To determine the content of chlorides in the given water sample/waste water sample.
7	To determine the concentration of Total Solids in the sample.
8	To determine the Chemical Oxygen Demand (COD) of given sample.
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15	Preparation/compilation and study of MSDS of certain chemicals used in your laboratory.

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Experiment No: Date:

STANDARDIZATION OF 0.01M EDTA SOLUTION

Aim: Standardization of 0.01M EDTA solution by using 0.01N $CaCO_3$.

Apparatus: Titration Set, Measuring Cylinder, Weigh Balance.

Chemicals: $CaCO_3$ powder, 0.01M EDTA solution, Ammonia buffer, EBT indicator.

Preparation of Chemicals:

0.01M EDTA
Weigh accurately 3.723g of disodium salt of EDTA. Dissolve it in distilled water and make up the solution to one later.

Buffer Solution
Dissolve 17.4 g of ammonium chloride in 142 ml of concentrated ammonia. Distilled water is then added to it to make up a required volume of 250 ml.

Erichrome Black-T Indicator
Dissolve 0.2 g of Erichrome Black T indicator in 15 ml of concentrated ammonia solution (or 15 ml of tri-ethanolamine) and 5ml absolute ethanol.

0.01M $CaCO_3$
Dissolve 0.2gm of $CaCO_3$ powder in 200ml water to get 0.01M CaCO3.

Procedure:

- Clean all the apparatus with distilled water.
- Fill the burette with 0.01M EDTA solution.
- Take 10ml of 0.01M $CaCO_3$ solution in conical flask.
- Add 5ml of ammonia buffer to the conical flask.
- Add 2-3 drop of EBT indicator to the conical flask solution. The colour of the solution becomes wine red.
- Run the burette solution to the conical flask till the solution turns sky blue.
- Note down the burette solution reading in the observation table.
- Repeat the process till two or three constant readings are obtained.
- Calculate the molarity using the burette reading obtained.

Observations

1. Burette : 0.01M EDTA
2. Pipette : 10ml 0.01M $CaCO_3$ solution
3. Indicator : EBT
4. Colour Change : Wine red to sky blue.

Chemical Reaction

Ca^{+2} + EBT ---------> [Ca^{+2} EBT] complex (wine red colour)

[Ca^{+2} EBT] + EDTA ---------> [Ca^{+2} EDTA] + EBT (stable sky blue complex)

Observation Table

Burette Reading	1st Reading	2nd Reading	3rd Reading	Average reading (B.R)
Initial	0	0	0	5.7
Final	5.9	5.7	5.7	
Difference	5.9	5.7	5.7	

Calculation

$M_1 V_1 = M_2 V_2$

Where, M_1 = Molarity of EDTA solution = x (unknown)

V_1 = Burette Reading

M_2 = Molarity of CaCo3 solution = 0.01M

V_2 = Volume of CaCo3 solution = 10ml

Therefore, M_1 = $\dfrac{M_2 V_2}{V_1}$

= _____

= ___

Result:

The molarity of EDTA solution used is _____.

Quiz:

1. What is EDTA? Write its structure.

2. In what form EDTA used"? Give structure.
3. Which indicator is employed usually in EDTA titrations? Give its IUPAC name and structure.
4. How does Eriochrome Black – T act as Indicator?

Marks: **Signature:**

Experiment No: Date:

TOTAL HARDNESS OF WATER

Aim: To determine the total hardness of water sample.

Apparatus: Titration Set, Measuring Cylinder, Weigh Balance.

Chemicals: Ammonium Chloride, Ammonium Hydroxide, EDTA, EBT, Magnesium Sulphate.

Preparation of Chemicals:

0.01M EDTA
Weigh accurately 3.723g of disodium salt of EDTA. Dissolve it in distilled water and make up the solution to one litre.

Buffer Solution
Dissolve 17.4 g of ammonium chloride in 142 ml of concentrated ammonia. Distilled water is then added to it to make up a required volume of 250 ml.

Erichrome Black-T Indicator
Dissolve 0.2 g of Eriochrome Black T indicator in 15 ml of concentrated ammonia solution (or 15 ml of tri-ethanolamine) and 5ml absolute ethanol.

0.01M $CaCO_3$
Dissolve 0.2gm of $CaCO_3$ powder in 200ml water.

1 N NaOH
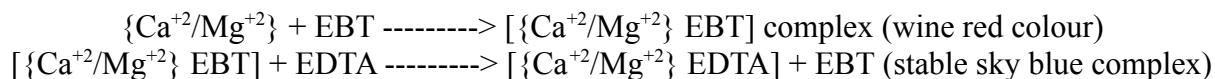Dissolve 10gm of NaOH pallets in 250ml water.

Procedure:

- Clean all the apparatus with distilled water.
- Fill the burette with 0.01M EDTA solution.
- Take 20 ml of water sample in conical flask with the help of a pipette.
- Add 5ml of ammonia buffer to the conical flask.
- Add 2-3 drop of EBT indicator to the conical flask solution. The colour of the solution becomes wine red.
- Run the burette solution to the conical flask till the solution turns sky blue.
- Note down the burette solution reading in the observation table.
- Repeat the process till two or three constant readings are obtained.
- Calculate the total hardness as $CaCO_3$ in mg/lt using the burette reading obtained.

Observations:

1. Burette : 0.01M EDTA
2. Pipette : 20 ml water sample
3. Indicator : EBT
4. Colour Change : Wine red to sky blue.

Chemical Reaction

$\{Ca^{+2}/Mg^{+2}\}$ + EBT ---------> [$\{Ca^{+2}/Mg^{+2}\}$ EBT] complex (wine red colour)
[$\{Ca^{+2}/Mg^{+2}\}$ EBT] + EDTA ---------> [$\{Ca^{+2}/Mg^{+2}\}$ EDTA] + EBT (stable sky blue complex)

Observation Table

Burette Reading	1st Reading	2nd Reading	3rd Reading	Average reading (B.R)
Initial	0	0	0	
Final				
Difference				

Calculation:

Volume of EDTA used : ml
Normality of EDTA used : N
Volume of water sample taken : ml
Equivalent weight of $CaCO_3$: 50

Total Hardness : Volume of EDTA * Normality * Eq. wt of $CaCO_3$ * 1000

Volume of sample taken

=

=

Result:

The total hardness of water sample is _____.

Quiz:

1. What is hardness of water?
2. What is temporary or carbonate hardness of water?
3. What is permanent or Non- carbonate hardness of water?
4. Why hard water does not lather with sop?
5. Which is the best method for hardness determination and why?

Marks: **Signature:**

Experiment No: Date:

CALCIUM HARDNESS OF WATER

Aim: To determine the calcium hardness of water sample.

Apparatus: Titration Set, Measuring Cylinder, Weigh Balance.

Chemicals: Sodium Chloride, 1N Sodium Hydroxide Ammonium Purpurate, EDTA.

Preparation of Chemicals:

0.01M EDTA
Weigh accurately 3.723g of disodium salt of EDTA. Dissolve it in distilled water and make up the solution to one litre.

Ammonium Purpurate
Weigh 0.5gm of ammonium Purpurate. Transfer it to a dry beaker. Weigh 100gm of sodium chloride and transfer it to beaker having ammonium Purpurate and mix well. Use it as a dry powder.

Sodium Hydroxide Solution (1N)
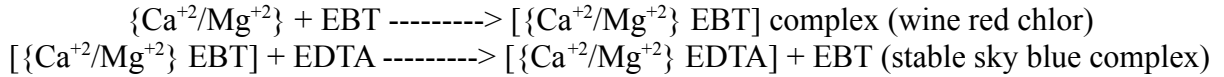Measure 4gm of NaOH and dissolve it in 100ml of distilled water.

Procedure:

- Clean all the apparatus with distilled water.
- Fill the burette with 0.01M EDTA solution.
- Take 20 ml of water sample in conical flask with the help of a pipette.
- Add 2ml of sodium hydroxide to the conical flask in order to maintain pH between 12 to 13.
- Add a pinch of ammonium Purpurate to the above solution. The colour of the solution becomes pink. The colour change is due to presence of calcium and magnesium hardness of water.
- Run the burette solution to the conical flask till the solution turns purple.
- Note down the burette solution reading in the observation table.
- Repeat the process till two or three constant readings are obtained.
- Calculate the calcium hardness of water.

Observations:

1.   Burette   :   0.01M EDTA
2.   Pipette   :   20 ml water sample
3.   Indicator   :   Ammonium Purpurate
4.   Colour Change   :   Pink to purple.

Chemical Reaction

$\{Ca^{+2}/Mg^{+2}\}$ + EBT ---------> [$\{Ca^{+2}/Mg^{+2}\}$ EBT] complex (wine red chlor)
[$\{Ca^{+2}/Mg^{+2}\}$ EBT] + EDTA ---------> [$\{Ca^{+2}/Mg^{+2}\}$ EDTA] + EBT (stable sky blue complex)

Observation Table

Burette Reading	1st Reading	2nd Reading	3rd Reading	Average reading (B.R)
Initial	0	0	0	9.2
Final	9.5	9.1	8.9	
Difference	9.5	9.1	8.9	

Calculation:

Volume of EDTA used   :   ml
Normality of EDTA used   :   N
Volume of water sample taken   :   ml
Equivalent weight of $CaCO_3$: 50

Calcium Hardness   : Volume of EDTA * Normality * Eq. wt of CaCo3 *
1000

Volume of sample taken

=   mg/lit as $CaCO_3$ equivalent.

Calcium present in sample   : Ca hardness in mg/lt as $CaCO_3$ * Mol. Wt of Ca

Mol. Wt of $CaCO_3$

=   mg/lit

Result:

The Calcium hardness of water sample is _____.

Calcium Ion present in sample is _____mg/lit.

Quiz:

1. How hardness of water is expressed?
2. What are various units of hardness and how are they related to each other?
3. What are soap? Why do not they form from lather with hard water?
4. How permanent hardness of water can be removed?
5. How can the hardness due to Mg^{2+} ion can be estimated?

Marks: **Signature**

Experiment No: Date:

DETERMINATION OF ACIDITY

Aim:

To determine acidity of a given water sample.

Theory:

Acidity in water is mainly due to the mineral acids and carbon dioxide. As such, it is the measure of the ability of water to neutralize bases (HCO^-_3, CO^-_2, OH^-).

Carbon dioxide acidity is due to presence of free CO_2 in ground and surface waters. Mineral acidity is due to the presence of HCl, H_2SO_4, HNO_3 and strong organic acids. It is expressed in terms of $CaCO_3$ equivalent of the hydroxyl ions neutralized.

Types of acidity:

(1) Mineral acidity
(2) CO_2 acidity

Carbon dioxide acidity is present in ground water and surface water from the hypolimnion of stratified lakes and reservoir due to oxidation of organic matter by bacteria.

Mineral acidity is present in many industrial wastes, particularly those of metallurgical industry and from the production of synthetic organic materials. Certain natural waters may also contain mineral acidity. The drainage from abandoned mines, lean ore – dumps and gob – piles will contain significant amount of sulfuric acid.

Most natural waters, domestic wastewater and many industrial wastes are buffered principally by a carbon dioxide-bicarbonate system. On the basis of the information it is customary to consider that all waters having pH lower than 8.5 contain acidity. Usually the phenolphthalein end point at pH 8.2 to 8.4 is taken as the reference point. The carbon dioxide (carbonic acid) alone will not depress the pH below a value of about 4. At pH 7 considerable carbon dioxide remains to be neutralized. It is concluded that neutralization of the strong acid is essentially complete at 3.7 to 4. Thus, from the nature of the titration curve for carbonic acid and for strong acids, it becomes obvious that the acidity of the natural water is caused by carbon dioxide or by strong mineral acids, the former being the effective agent in waters having pH value greater than 4 and the latter the effective agent in water with pH less than 4. As shown in figure.

Sources and nature of acidity:

Type of acidity is of importance in ordinary water & wastewater analysis & pH ranges in which they are significant.

Carbon dioxide is a normal component of all natural waters.

Surface waters are constantly absorbing or giving up carbon dioxide to maintain equilibrium with the atmosphere. The amount that can exist at equilibrium is very small because of the low partial pressure of carbon dioxide in the atmosphere.

Ground waters & waters from the hypolimnion of stratified lakes & reservoirs often contain considerable amounts of carbon dioxide. These concentration results from bacterial oxidation

of organic matter with which the water has been in contact & under this condition the carbon dioxide is not free to escape to the atmosphere.

Carbon dioxide is an end product of both aerobic & anaerobic bacterial oxidation; so its concentration is not limited by the amount of dissolved oxygen originally present.

Carbon dioxide determinations are particularly important in the field of public water supplies. In the development of new supplies, it is an important factor that must be considered in the treatment method and the facilities needed. Many underground supplies require treatment to overcome corrosive characteristics resulting from carbon dioxide. The amount present is in important factor in determining whether removal by aeration or simple neutralization with lime or sodium hydroxide will be chosen as the treatment method. The size of equipment, a chemical requirement storage spaces and cost of treatment all depend upon the amounts of carbon dioxide present. Carbon dioxide is an important consideration in the chemical requirements for lime or lime soda ash softening.

Environmental significance of carbon dioxide and mineral acidity:

1. Acidity interferes in the treatment of water (as in softening).
2. It corrodes pipes (zinc coating of G.I. pipes got dissolved).
3. Aquatic life will be affected.
4. P^H is critical factor for bi-chemical reaction.
5. Waters contain mineral acidity are so unpalatable.
6. Waters having acidity more than 50 mg/l cannot be used in R.C.C works.

Application of acidity data in Environmental Engineering practice:

1. The amount of CO_2 present is an important factor in determining whether removal by aeration or simple neutralization with lime or sodium hydroxide will be chosen as the treatment method.
2. The size of equipment, chemical requirement storage spaces and cost of treatment all depend upon amount CO_2 present.
3. CO_2 is an important consideration in estimating chemical requirement for lime soda-ash softening processes.
4. Most industrial wastes containing mineral acidity must be neutralized before they are subjected to biological treatment or direct discharge into watercourses or sewers. Quantities of chemical, size of chemical feeders, storage space and costs are determined from the laboratory data of acidity.

Determination of Acidity:

Field Method:

The titration procedure has many advantages and is sufficiently accurate for all practical purposes.

Methyl Orange Acidity :(Mineral Acidity)

All natural waters and most industrial waters that have a pH below 4 contain minerals or methyl orange acidity. Mineral acids are essentially neutralized by the time the pH has been

raised to about 3.7 and a colour pH indicator is normally used where a pH meter is not available. While methyl orange was formerly used for this purpose, bromophenol blue is now recommended as it has a sharper colour change at pH 3.7. Results are reported in terms of methyl orange acidity expressed as $CaCO_3$. Since $CaCO_3$ has an equivalent weight of 50, N/50. NaOH is used as the titrating agent so that 1 ml is equivalent to 1 mg of acidity.

Phenolphthalein Acidity:

Occasionally, it is desirable to measure the total acidity resulting both from mineral acids and from weak acids in the sample. Since, most weak acids are essentially neutralized by titration to pH 8.3 either phenolphthalein or Meta cresol purple indicators can be used for this titration. When heavy metal salts are present, it is usually desirable to heat the sample to boiling and then carry out the titration to be completed more rapidly. Again N/50 NaOH is used as the titration agent and results are reported in terms of phenolphthalein acidity expressed as $CaCO_3$.

Principle:

The mineral acids present in the sample which are contributing mineral acidity can be calculated by titrating or neutralizing samples with strong base NaOH to P^H 4.3. The CO_2 and bicarbonates (carbonic acid) present and contribute CO_2 acidity in the sample can be neutralized completely by continuing the titration to pH 8.2.

Reagent:

Carbon dioxide free water
Standard sodium hydroxide titrant, 0.02N
Hydrogen peroxide, H_2O_2, 30%
Bromophenol blue indicator solution pH 3.7 indicator or methyl orange indicator
Meta cresol purple indicator solution, alcoholic, pH 8.3 indicator or
Phenolphthalein indicator solution, alcoholic, pH 8.3 indicator
Sodium thiosulphate, 0.1 M

Interference:

Colour, turbidity, iron, aluminium or manganese and residual chloride are prime sources of interference. Colour and turbidity can be avoided using potentiometer titration. Residual chlorine can be removed by adding sodium thiosulphate. Iron, aluminium and manganese is prevented by the addition of Na – K tartrate.

Apparatus:

Titration vessel
Pipettes
Volumetric Flasks, 1000-200-100 ml
Burettes borosilicate glass, 50-25-10 ml
Polyolefin bottle, 1-L

Procedure:

(1) Take 100 ml of the given sample in an Erlenmeyer flask.

(2) Add 1 drop of 0.1 N sodium thiosulphate solution to remove the residual chlorine if present.
(3) Add 2 drops of methyl orange. The sample turns pink.
(4) Proceed with titration until the colour changes to yellow.
(5) Note down the volume of the NaOH added (V_1).
(6) Take another conical flask containing 100 ml of water sample, add 2 or 3 drops of phenolphthalein.
(7) Proceed with titration until the sample turns pink.
(8) Note down the total volume of NaOH added (V_2).

Observations and Results:

Sr. No.	Sample details	Volume of sample (ml)	Phenolphthalein Indicator			Methyl orange Indicator		
			Initial burette Reading (ml)	Final burette Reading (ml)	NaOH used (ml)	Initial Burette (ml)	Final Burette (ml)	NaOH used (ml)

Calculations:

Mineral acidity due to mineral acids (as $CaCO_3$) (mg/l)

$$= \frac{V_1 \times N \times 1000 \times 50}{\text{ml of sample taken}}$$

CO_2 acidity due to CO_2 (as $CaCO_3$) (mg/l)

$$= \frac{V_2 \times N \times 1000 \times 50}{\text{ml of sample taken}}$$

Total acidity as ($CaCO_3$) (mg/l)   = Mineral acidity + CO_2 acidity =

Results:

Mineral acidity (mg/l)   = CO_2 acidity (mg/l)   =

Total acidity as ($CaCO_3$)   = Mineral acidity + CO_2 acidity =

Discussion of results:

Quiz:

1. What is the significance of determining acidity in water?
2. Why the two indicators namely phenolphthalein and methyl orange are selected for determining acidity?
3. Differentiate between acidity and alkalinity of water?
4. Define acidity of water?
5. Discuss the roll phenolphthalein as an acid base indicator.

Marks: **Signature:**

Experiment No: Date:

DETREMINATION OF ALKALINITY

Aim:

To determine the alkalinity of a water sample.

Theory:

Alkalinity is a measure of the ability of water to neutralize acids. It is expressed in terms of $CaCO_3$ equivalent of the hydrogen ions neutralized. Alkalinity of water is due to presence of the following:
(1) Bicarbonates, carbonates, hydroxides of sodium, potassium, calcium, and magnesium.
(2) Salts of week acids and strong bases as:
    (a)   Borates, silicates, and phosphates
    (b)   Salts of organic acids and humic acids
    (c)   Salts of acetic, propionic and hydrosulphuric acids.
(3)   Algae utilize the free and combined carbon dioxide present in natural waters during Photo synthesis.

However, the major portion of the alkalinity in the natural water is caused by carbonates, bicarbonates and hydroxides, which may be ranked in order to their association with high pH values as shown in figure.

Boiler waters always contain carbonate and hydroxide alkalinity, chemically treated waters (lime or lime soda ash softening waters) will be alkaline due to presence of carbonates and excess hydroxides. High alkalinity in natural waters will favour the growth of producers (algae and planktons).

Public health significance:

Highly alkaline waters are usually unpalatable and consumers tend to seek other supplies.
Chemically treated waters sometimes have rather high pH values, which have met with some objection on the part of consumers.
Large amount of alkalinity impart a bitter taste to water.
The principal objection of alkaline water is the reactions that can occur between alkalinity and certain cations in waters. The resultant precipitate can foul pipes and other appurtenances of water distribution systems.

Application of alkalinity data in Environmental Engineering practice:

(1) Chemical coagulation of water and waste water-
    To neutralize acids produced during flocculation, the sample should be alkaline as otherwise further floc formation (either Al $(OH)_3$ or Fe $(OH)_3$) slowly ceases.

(2) Water softening-
    To find out the quantity of lime and soda ash required for the removal of hardness,

    alkalinity should be found out.

(3) Corrosion control-
To control the corrosion dye to acids, natural waters are rendered to alkaline.

(4) Effluents of waste water-
Wastewater containing excess caustic (hydroxide) alkalinity are not to be discharged into

natural water bodies or sewers.

Excess alkalinity in water is harmful for irrigation, which leads to soil damage and reduces crop yields. Water having an alkalinity content of less than 250 mg/l is desirable for domestic consumption.

Method of determination:

Alkalinity is generally determined by titration method.

Principle:

Alkalinity can be obtained by neutralizing OH^-, CO^-_3 and HCO_3^- with standard H_2SO_4. Titration to pH 8.3 or decolourization of phenolphthalein indicator, will show complete neutralization of OH^- and half of CO_3^{--}, while to pH 4.4 or sharp change from yellow to pink of methyl orange indicator will indicate total alkalinity, i.e., OH^-, $CO3^{--}$ and HCO_3^-

Interference factors:

Interference caused by colour, turbidity of free chlorine has already been discussed in acidity test. In addition, absorption or loss of CO_2 during or after sample collection can falsify the results.

Apparatus:

Burette
Conical flask
Pipettes etc.

Reagents:

Standard H_2SO_4 (0.1 N)
Phenolphthalein Indicator
Methyl orange indicator
CO_2 free distilled water
Sodium thiosulphate (0.1 N)

Procedure:

(1) Take 100 ml of given sample in a conical flask
(2) Add one drops of sodium thiosulphate (0.1N) to remove the free residual chlorine, if present.
(3) Add two drops of phenolphthalein indicator, the sample turns pink.
(4) Run down 0.1 N standard H_2SO_4 till the solution turns to colourless.
(5) Note down the volume of H_2SO_4 added (V1).

(6) Add two drops of Methyl Orange indicator, the sample turns to yellow.
(7) Resume titration till the colour of solution turns to pink.
(8) Note down the volume of H_2SO_4 added (V2).
(9) Normality test for 0.1 N standard H_2SO_4
   Take 20 ml of 0.1 N standard H_2SO_4 in a conical flask and titrate against 0.5 N Na_2CO_3 and note down the reading.
   N x 20 ml = 0.5 x titrant consumed in ml
   N = _____

Observations and Results:

Sr. No.	Sample details	Volume of sample (ml)	Phenolphthalein			Methyl orange		
			Initial burette Reading (ml)	Final burette Reading (ml)	H_2SO_4 used V1(ml)	Initial Burette (ml)	Final Burette (ml)	H_2SO_4 used V2(ml)

Calculations:

Phenolphthalein alkalinity (P) as mg $CaCO_3$ / l
= V1 x normality of H_2SO_4 x 1000 x 50
   Volume of sample taken

Total alkalinity (T) as $CaCO_3$ mg/l
= V2 x normality of H_2SO_4 x 1000 x 50
   Volume of sample taken

Value of P and T	Alkalinity due to		
	OH^-	CO_3^{--}	HCO_3^-
P = 0	O	O	T
P<1/2T	O	2P	T-2P
P=1/2T	O	2P	O
P>1/2T	2P-T	2T-2P	O
P=T	T	O	O

Results:

Hydroxide alkalinity (mg/l) =
Carbonate alkalinity (mg/l) =

Bicarbonate alkalinity (mg/l) =

Discussion of results:

Quiz:

1. What is the significance of determining alkalinity in water?
2. Why the alkalinity of irrigation water is determine?
3. On what factor the choice of indicator for a particular acid-base titration depends?
4. Why a titration of weak acid against a weak base is avoided usually?
5. Give an example of the acid – base titration when the pH value is less than 7 at the end point.

Marks: **Signature:**

Experiment No: Date:

DETERMINATION OF CHLORIDES

Aim:

To determine the content of chlorides in the given water sample/waste water sample.

Theory:

Chloride is present in all natural waters at greatly varying concentration depend on the geochemical conditions. Chlorides in natural waters can be attributed to leaching of chloride containing rock and soils, discharges of effluents from chemical industries, ice-cream plant effluents, edible oil mill operations, sewage disposal, irrigation drainage, contamination from refuse leachates and sea water intrusion in coastal regions. Each of these sources may result in local contamination of both surface and ground water.

Large amounts of chlorides reach to the receiving water through leaching of domestic sewage. A man discharges 8 to 10 gm per day of NaCl through both urine and excreta. As such, domestic sewage contains more chlorides than domestic water supply. Due to this chloride can often serve as a chemical – pollution indicator of domestic sewage contamination when considered together with other parameters and a natural geological origin does not apply.

When chlorides concentration of 250 mg/L is present along with sodium ions, a salty taste can be observed. The salty taste may be absent in waters in absence of sodium ions, even concentration of chlorides is as high as 1000 mg/L.

Chlorides are highly soluble with most of naturally occurring cations expect with silver. Chlorides can only be removed by reverse osmosis process and electrolysis. Seawater has chlorides content of 19000 to 20000 mg/L.S

Chlorides occur in all natural waters in varying concentrations. The chloride content normally increases with the increase in mineral content. Upland and mountain supplies are low in chlorides while river and ground waters have a considerable amount. Sea and ocean waters are exposed to partial evaporation of natural waters that flow into them. So the chloride levels are very high.

Chlorides gain access to the natural waters due to the solvent power of water, which dissolves chloride from topsoil. Oceans and seawaters invade rivers that drain into them. The salt water being denser flows upstream under the fresh water flowing downstream. So there is a constant mixing of salt water in the upper areas. Ground waters in areas adjacent to the oceans are in hydrostatic balance with seawater. Water used for irrigation is lost to the atmosphere through evapotranspiration, leaving the salts, originally present in the water behind in the soil. When fresh water is added to the fields, saline water is returned (due to the salts present in the soil) So the chloride content increases in that water source in which it is discharged. Sodium chloride consumed by humans through food and water passes unchanged through the digestive system. So an average of 6 g of chloride per person, per day is added to the municipal wastewater. Industrial processes also increase the chloride content in wastewater.

Environmental Significance:

1. Chlorides at concentrations above 250 mgs/1 give an objectionable salty taste to water.
2. Before the development of bacterial testing procedures, chemical tests for chlorides and various forms of nitrogen. Served as the basis for detecting contamination of ground water by waste water.
3. Evapotranspiration increases the chloride and salinity of soil. Making it difficult of the crops to take up water due to osmotic pressure differences between water outside and water with in the plant.
4. High chloride content may harm metallic pipes and other such structures.

Application of Chloride data in Environmental Engineering practice:

1. Chloride data helps in selection of water supplies for human, industrial and agricultural use.
2. It helps in determining the type of desalting apparatus to be used.
3. It helps in determining whether seawater has intruded ground water. If so such water need not be pumped out.
4. It helps in determining whether the salt water brines and industrial waste waters can be discharged in a water sources or not.
5. Chlorides interfere with the determination of COD. So depending on the amount of chloride, mercuric sulphate has to be added during COD to remove chloride interference.

Argentometric (Mohr) Method for Chloride determination:

Principle:

Chloride ion is determined by Mohr's method, titration with standard silver nitrate solution in which silver chloride is precipitated at first. The end of titration is indicated by formation of red silver chromate from excess $AgNO_3$ and potassium chromate used as an indicator in neutral to slightly alkaline solution.

$AgNO_3 + Cl^-$ $AgCl + NO_3^-$
                                  (White)

$2AgNO_3 + K_2CrO_4$ $Ag_2CrO_4 + 2\ KNO_3$
                                  (Red)

Interferences:

1. Bromide, iodide and cyanide, Sulphide, thiosulphate and phosphates are prime sources of interference can be removed by treatment with hydrogen peroxide.
2. Orthophosphate in excess of 25 mg/l, interferes by precipitating a silver phosphate.

Apparatus:

Conical Flask
Burette
Pipette

Measuring Cylinder
Beaker

Reagents:

Chloride free distilled water
0.0141 M Silver Nitrate Solution
0.0141 M Sodium Chloride Solution
Potassium Chromate Indicator solution

Procedure:

1. Take 100 ml of water sample
2. Adjust its P^H to be between 7 and 8 either with sulfuric acid or sodium hydroxide solution. Otherwise, AgOH is formed at high P^H level or CrO_4^{-2} is converted $Cr_2O_7^{-2}$
3. Add 1 ml of K_2CrO_4 indicator solution to get light yellow colour.
4. Titration with standard $AgNO_3$ solution till colour change from yellow to brick red.
5. Note down the volume of silver nitrate added (A).
6. If more quantity of potassium chromate is added, Ag_2CrO_4 may form too soon or not soon enough.
7. For better accuracy, titrate distilled water in the same manner.
8. Note down the volume of silver nitrate added for distilled water (B)

Observation Table:

Sr. No.	Source of sample	Volume of the Sample	Initial Burette Reading	Final Burette Reading	Volume of $AgNO_3$ used
1.					
2.					
3.					
4.					
5.					
6.					

Calculations:

Concentration of Chloride in mg/l

$$= \frac{(A - B) * \text{Normality of } AgNO_3 * 35.46 * 1000}{\text{Volume of sample taken}}$$

Result:

Chloride content of the given water/waste water sample is _____mg/1

Discussion of results:

Quiz:

1. Name the sources of chlorides in water.
2. Why $AgNO_3$ combine first with chloride ions in the water and not with K_2CrO_3?
3. What is the principal of Mohr's method?
4. How is the solution neutralised if it is acidic or basic?
5. Discuss limitations of Mohr's method.

Marks: **Signature:**

Experiment No: Date:

DETERMINATION OF TOTAL SOLIDS

Aim:

To determine the concentration of Total Solids in the sample.

Theory:

The matter that remains upon evaporation and drying a water sample at 103° C to 105° C is the total solid material present in the sample. The residue remaining represents only those materials present in the sample that has a negligible vapour pressure at 105° C. Determination of the amount of dissolved and un dissolved solids is accomplished by making tests on the filtered and unfiltered portions of samples. The total solids content of potable water usually range from 20 to 1000 mg/lit and as a rule, hardness increases with total solids. Volatile and fixed solids are determined by carrying out combustion in a muffle furnace where the temperature can be accurately controlled. If ignitions are properly performed, the weight loss incurred is a reasonably accurate measure of organic matter and the residue remaining represents the ash or fixed solids. A rapid estimation of the dissolved solids contents of the water sample can be obtained by specific conductance measurements.

Application of Total Solids data in Environmental Engineering Practice:

Total solids determination is useful for water softening. Corrosion control is done by pH adjustment, which depends on total solids concentration.
Suspended solids and volatile suspended solids are used to evaluate the strength of sewage and industrial wastes in the primary sedimentation tanks. In stream pollution control the removal of suspended solids is as important as BOD removal. Total and volatile solids tests are more applicable to sludge. They are used in the design and operation of sludge digestion, vacuum filtration and incineration units.

Principle:

Total solids are determined as the residue left after evaporation and drying of the unfiltered sample.

Apparatus:

Evaporation dishes (Pyrex, porcelain- 103^0C or platinum, silica – 600 ^{0}C) or Borosil beakers, Hot air oven, desiccators, analytical balance.

Procedure:

1. A clean porcelain dish is ignited in a muffle furnace and after partial cooling in the air, it is cooled in a desiccator and weighed.
2. A 100 ml of well mixed sample (graduated cylinder is rinsed to ensure transfer of all suspended matter) is placed in the dish and evaporated at 100 0C on water bath, followed by drying in oven at 103° C to 105° C at 1 hour.
3. After drying. Cool the dish in the desiccator and weigh as the final weight.
4. The increase in the weight indicates in the total solids.

Observation Table and Results:

Sr. No.	Sample Details	Volume Of Sample	Weight of empty Crucible	Weight of dried Crucible	Difference In Wt. (g)	Total solids (mg/lit)	Average

			(Initial Wt. g)	(Final Wt. g)			
	Tap Water						
1							
2							
3							
	Waste water						
4							
5							
6							

Calculation:

Total solids (mg/lit) = $\dfrac{\text{Difference in weight} * 10^6}{\text{Volume of sample}}$

Result:

Total solids content of the given water/waste water sample is _____mg/1

Discussion of results:

Quiz:

1. How did you find total solid concentration?
2. What percentage of waste water is solids?
3. What is meant by total solids?
4. What are total solids in a water body?
5. How do you calculate total solids in milk?

Marks: **Signature:**

Experiment No: Date:

CHEMICAL OXYGEN DEMAND

Aim:

To determine the Chemical Oxygen Demand (COD) of given sample.

Theory:

Chemical Oxygen Demand (COD) is the oxygen required for the chemical oxidation of organic matter by strong chemical oxidant ($K_2Cr_2O_7$) under acidic condition. The degree of oxidation depends upon the type of substance, pH. value, temperature, reaction time and the concentration of oxidizing agent.

The main disadvantage of this test is that oxygen is also consumed by oxidation of inorganic substances such as nitrites, chlorides, sulphides, reduced metal ions. Also some of the organic materials like amino acids, ketones, or saturated carboxylic acids, benzene, pyridine etc are not oxidized by dichromate. Consequently this test is a poor measure of strength of organic wastes unless all these factors are considered.

One of the chief limitations of COD test is its inability to differentiate between biologically oxidizable and biologically inert organic matter. In addition it does not provide any evidence of the rate at which biologically active material would be stabilized under condition that exist in nature.

The COD values are greater than BOD values and may be much greater when significant amounts of biologically resistant organic matter is present.
This test may be quite useful as compared to BOD values for estimating strength of certain industrial wastes both organic as well as inorganic which contain toxic chemicals.

The major advantage of COD test is the short time required for evaluation. The determination can be made in about three hours rather than five days required for measurement of BOD.

Practical Relevance of Experiment and Application of Data:

COD data can often be interpreted in terms of BOD values after sufficient experience has been accumulated to establish reliable correlation factors. The COD test is used extensively in the analysis of industrial waste. The test is widely used in the operation of treatment facilities because of the speed with which the result can be obtain. COD data is used in conjunction with the BOD test. The COD test is helpful in indicating toxic conditions and the presence of biologically resistant organic substances.

Application of COD Data in Environmental Engineering Practice:

1. The COD test is used extensively in the analysis of industrial wastes.

2. It is particularly useful in designs to determine and control the losses in sewer systems.
3. This test is widely used in place of BOD test in the operation of treatment facilities because of the speed with which the results can be obtained.
4. It is useful to assess the strength of the wastes which contain toxins and biologically resistant organic substances.
5. The ratio of BOD to COD is useful to assess the amenability of the waste for biological treatment
6. BOD / COD > 0.8 indicates highly biodegradable waste and BOD / COD < 0.3 indicates biologically inert waste.

Principle:

The organic matter present in the sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 and H_2O. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with Standard Ferrous Ammonium Sulphate as titrant and Ferroin as indicator. The colour change is from green to red wine. The dichromate consumed gives the oxygen required for the oxidation of the organic matter.

Apparatus:

1. Reflux apparatus, consisting of 250 ml. Erlenmeyer flask or standard glass tubes with ground glass neck or equivalent condenser to fit within the ground glass neck of tubes of flask.
2. Heating plate to produce at 9 watts/sq. in. of heating surface a equivalent of insure adequate boiling of the contents of the refluxing flasks.
3. Glass beads.
4. Titration flask.
5. Pipette and Burette.

Reagents:

1. Standard potassium dichromate solution 0.25 N
2. Standard Ferrous Ammonium Sulphate titrant
3. Sulfuric acid reagent (H_2SO_4)
4. Silver sulphate ($AgSO_4$)
5. Mercuric sulphate ($HgSO_4$)
6. Ferroin indicator

Procedure:

1. Place 0.4 gm $HgSO_4$ in the reflux flask.
2. Add 20 ml of sample (or any amount of sample diluted to 20 ml).
3. Add 10 ml of standard potassium dichromate solution and several preheated glass bends in refluxing flask.

4. Slowly mix 30 ml of conc. Sulfuric acid containing silver sulphate to the flask
5. Connect flask with condenser tube.
6. Mix the contents thoroughly before heating.
7. Reflux the sample for a minimum period of 2 hours.
8. Allow it to cool down and wash condenser with distilled water.
9. Dilute the sample with distilled water to make up150 ml and cool down to room temperature.
10. Titrate excess dichromate with standard ferrous ammonium sulphate using 2 to 3 drops of Ferroin indicator.
11. The end point of titration is indicated by colour change from blue green to reddish brown.(even though the blue green colour may reappear within minutes)
12. Reflux the blank in the same manner using distilled water instead of sample.

Note: For an unknown sample to decide the amount of sample, take HgSO4, 10 ml $K_2Cr_2O_7$, and 20 ml distilled water and 30 ml conc. Sulfuric acid containing silver sulphate to the flask. Go on adding sample to this flask until the clear yellow colour of $K_2Cr_2O_7$ turns turbid.

Observation Table:

Sr. No.	Sample description	ml of ferrous ammonium sulphate used			COD mg /lit
		Initial reading	Final reading	Difference (ml)	

Calculation:

COD of sample in mg/lit = $\dfrac{(A-B) \times N \times 8000}{\text{ml of sample taken}}$

Where, A = ferrous ammonium sulphate used for blank
B = ferrous ammonium sulphate used for sample
N = Normality ferrous ammonium sulphate used

Result:

1. COD of sample _____ = mg/lit.
2. COD of sample _____ = mg/lit.
3. COD of sample _____ = mg/lit.

Discussion of result:

Quiz:

1. What is chemical oxygen demand?
2. Discuss the significance of COD determination?
3. What is Ferroin indicator and how it is prepared?
4. Name the impurities which may interfere in COD determination.
5. How you define the pollution strength of waste water and Industrial waste?
6. Correlate BOD with COD.

Marks: **Signature:**

Experiment No: Date:

DETERMINATION OF DISSOLVED OXYGEN

Aim:

To find out the quantity of Dissolved Oxygen present in the given sample.

Theory:

The presence of oxygen is essential for the survival of aquatic life in water.

Non-polluted surface waters are normally saturated with dissolved oxygen.

A rapid fall of DO level in river waters is one of the first indications of organic pollution.

Thus, it is one of the important parameters for assessing the quality of water bodies and plays essential role in water pollution control activities.

The major input of dissolved oxygen to natural water is from the atmosphere and photosynthetic reaction where the algae and phytoplankton production is high, the over-saturation of oxygen can occur during daytime.

The solubility of oxygen in water depends on the temperatures, pressure, altitude etc.

The solubility of oxygen decreases with increases in temperature in fresh water.

The solubility of atmospheric oxygen in fresh water is from 14.6 mg/l at 0 °C to about 7 mg/l at 35 °C under atmospheric pressure.

Environmental Significance:

Drinking water should be rich in D.O. for good taste. Aerobic bacteria thrive when free oxygen is available in plenty. Aerobic condition does prevail when D.O. is sufficient and product is stable and does not foul smell. Minimum 4 to 5 mg/l D.O. is desirable for the survival of the aquatic life. Higher temperature, biological impurities, ammonia, nitrites, ferrous iron, hydrogen sulphide and organic matter reduces D.O. level.

Application of DO data in Environmental Engineering Practice:

1) To assess quality of raw water and to keep a check on stream pollution.
2) D.O. test is the basis for B.O.D. test which is an important parameters to know the pollutional strength of water.
3) D.O. test is necessary for all biological waste water treatment to control the rate of aeration.
4) D.O. is important in the corrosion of iron and steel. D.O. test is used to control oxygen in boiler feed water.

Amongst the experimental methods, mainly two are very popular.
1. Analytical Method
2. Electrode / Probe method

1) **Analytical Method**: The analytical method and its modification to determine dissolved oxygen is known as Wrinkler or Iodometric method. The oxidation potential of oxygen is used in oxidizing Mn^{-2} to higher state of valency under alkaline conditions and this manganese in higher state of valency is capable of oxidizing I to free I_2 under acidic conditions. Thus, the amount of free iodine is measured with standard Sodium Thiosulphate solution and is equivalent to the amount of dissolved oxygen present.

Above stages can be summarized in order of the reaction as follows:

Mn^{2+} + 2OH⁻ + 1/2O_2 $\quad\quad\quad\quad$ MnO_2 + H_2O

MnO_2 + 2I⁻ + 4H⁺ $\quad\quad\quad\quad\quad\quad$ Mn^{2+} + I_2 + 2H_2O

Modification in the process is introduced for removing interference caused by nitrates which are likely to be in the sample. The interference is caused by nitrates by converting I⁻ to free I_2 which is interpreted as dissolved oxygen. Adding sodium azide in the alkaline reagent this interference is prevented by converting NO_2 to N_2O with the help of ammonia formed by the azide under acidic condition.

Principle:

Oxygen present in sample oxidizes the divalent manga nous to its higher valency forming brown colour precipitates of hydrated oxide after addition of NaOH and KI. Upon acidification, manganese reverts to back its divalent state and liberates iodine from KI equivalent to D.O. content in the sample. The liberated iodine is titrated against $Na_2S_2O_3$ (0.025 N), using starch as an indicator. The end point of the titration is marked by end point from blue to colourless. If oxygen is absent in the sample, the $MnSO_4$ react with the alkali to form white precipitates of Mn (OH) $_2$.

Apparatus:

1. BOD Bottles,
2. 250 ml reagent bottles with stopper,
3. Burettes,
4. Pipettes,
5. Measuring cylinders.

Reagents:

1. Manga nous sulphate solution,
2. Alkaline iodide azide reagent,
3. Concentrated Sulphuric acid,
4. Starch Solution,
5. Sodium Thiosulphate 0.025N.

Procedure:

1. Take the sample into 250 ml BOD bottle or reagent bottle with a stopper.
2. Add 2 ml of manga nous sulphate solution followed by 2 ml of alkaline iodide azide reagent. Both of these are to be added well below the surface of the liquid.
3. Stopper with care to exclude bubbles and mix by inverting the bottle several times.
4. When the precipitates settle, remove the stopper carefully and add 2 ml of conc. sulphuric acid immediately by allowing the acid to run down the neck of the bottle.
5. Restore the bottle stopper and mix the contents by gentle inversion until solution is completely mixed and the precipitates are dissolved completely.
6. From this solution, take 203* ml of sample and titrate against 0.025 N Sodium Thiosulphate from a burette until it gives pale yellow colour.
7. Add 1ml of starch solution now, it will give blue colour.
8. Continue the titration until the blue colour disappears.

*Note: As 1 ml of 0.025N sodium thiosulphate solution is equivalent to 0.2 mg oxygen, the ml. of sodium thiosulphate used is equivalent to the mg / lit of dissolved oxygen if a volume equal to 203 ml of origin sample is titrated. This avoids any further calculations.

Observation Table:

Sr. No.	Type of sample	Initial Reading	Final Reading	Difference	D.O.
		ml	ml	ml	mg/lit

Result:
1. For Sample _____ Amount of dissolved oxygen = _____ mg/lit.
2. For Sample _____ Amount of dissolved oxygen = _____ mg/lit.

3. Electrode Method:

The dissolved oxygen electrode consists of inert metal cathodic and silver anode. These are electrically connected by KCL or other electrolyte solution. The complete assembly is separated from the sample by means of gas permeable membrane made up of polyethylene or Teflon. When a potential of about 0.5 to 0.8 volt is applied across the anode and cathode, any oxygen which passes through membrane will be reduced at cathode, causing the current to flow. The current value is proportional to the amount of oxygen in the sample. This gives rapid determination of dissolved oxygen in the sample.

Procedure:

1. Keep the MODE switch at set 20.
2. Switch on the instrument. Adjust the set 20 control.
3. Switch the MODE switch at ZERO and adjust the ZERO control for 0.0 (this is the zero setting of the unit.).
4. Attach the prepared DO probe to the probe connector.
5. Allow 15 minutes for polarization of the probe.
6. Depolarize whenever the instrument is off.
7. Calibrate the instrument and place the PROBE in the sample to be measured.
8. The sample should be continuously stirred by the stirrer during the measurements to prevent depletion of DO near the membranes of the PROBE.
9. Set the SALINITY control to the salinity value of the sample under test.
10. Allow sufficient time for probe to stabilize.
11. Read the D.O. concentration.

Observation Table:

Sr. No.	Type of sample	D.O.

Results:
1. For Sample _____ Amount of dissolved oxygen = _____ mg/lit.
2. For Sample _____ Amount of dissolved oxygen = _____ mg/lit.

Discussion of results:

Conclusion:

Quiz:
1. What is the effect of the presence of oxidising impurities like NO_2^- and Fe^{3+} (If not removed) on the D.O.results?
2. Discuss azide modification?
3. How does the presence of bubble inside incubator bottle affect the D.O. result?
4. Write down different reactions taking place while determining D.O.?
5. How do reducing impurities such as Fe^{2+}, SO_3^{2-}, S^{2-}, etc. effect the D.O.results?

Marks: **Signature:**

Experiment No: Date:

BIOCHEMICAL OXYGEN DEMAND

Aim:

To determine BOD of given sample.

Theory:

Biochemical Oxygen demand (BOD) is usually defined as the amount of oxygen required by micro-organisms for stabilizing carbonaceous biodegradable organic matter under specified time and temperature conditions.[27 ° C for 3 days or 20 ° C for 5 days]

Organic matter   + O_2 Micro-Organism CO_2 + New Bacteria   + H_2O
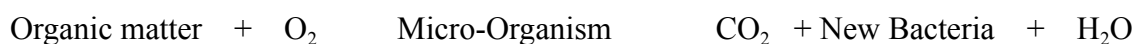
The BOD test is used to determine the pollutional strength domestic and industrial wastes in terms of the dissolved oxygen that the micro-organisms will require if discharged into natural water courses in which aerobic conditions exits. Hence this parameter is of utmost importance in stream-pollution control activities. It is also important in regulatory work and in studies designed to evaluate purification capacity of receiving bodies of water.

The BOD test involves measurement of oxygen consumed by living organisms (mainly Bacteria) while utilizing the organic matter present in wastewater. In order to make the test quantitative, the samples must be protected from the air to prevent the reaeration as the dissolved oxygen level diminishes. Because of the limited solubility of oxygen in water, about 9 mg/lit at 20 ^{0}C, strong wastes must be diluted to ensure that dissolved oxygen will be present throughout the period of the test.

It is extremely important that the environmental condition must be suitable for all living organisms to function in an unhindered manner at all times. To ensure this toxic substance must be absent and all the nutrients such as nitrogen, phosphorus and certain trace elements that are needed for bacterial growth must be present. If not naturally present in the sample they are added artificially in the form of dilution water.. Biological degradation of organic matter under natural conditions is brought by a diverse group of organisms that carry the oxidation essentially to completion i.e. almost entirely to CO_2 and water. Therefore it is important that a mixed group of organisms commonly called "Seed" is present in the test.

The BOD test may be considered as a wet oxidation producer in which the living organisms serve as the medium for oxidation of the organic matter to CO_2 and water. A quantitative relationship exists between the amount of oxygen required to convert a definite amount of any given organic compound to CO_2 water and ammonia. This can be represented by the following generalized equation:

$C_nH_aO_bN_c$ + $[n + a/4 - b/2 - 3/4c] O_2$ $nCO_2 + [a/2-3/2c] H_2O$ + cNH_3

On the basis of the above relationship, it is possible to intercept BOD data in terms of organic matter, as well as the amount of oxygen used during its oxidation. This concept is fundamental in understanding the rate at which BOD is exerted.

The oxidation reactions involved in this BOD test are a result of biological activity, and the rate at which the reactions proceed is governed to a major extent by population number and temperature. Temperature effects are held constant by performing the test at 20^0C.

Theoretically an infinite time is required for complete biological oxidation of organic matter but for all practical purposes, the reaction may be considered complete in 20 days. However, a 20 day period is too long to wait for results in most instances.

It has been found by experience that a reasonably large percentage of the total BOD (about 70%) is exerted in 5 days incubation period. It should be remembered, therefore that 5 day. BOD value represents only a portion of the total BOD. The exact percentage depends upon the character of the "seed" and the natural of the organic matter and can be determined only by experiment.

In the case of domestic and many industrial wastewaters, it has been found that the 5 days BOD value is about 70 to 80 % of the total BOD. This 5 day period brings out oxidation of almost all the carbonaceous organic matter present and after this incubation period oxidation of ammonical organic matter starts. Thus the 5 day incubation period was selected also to minimize interference from oxidation of ammonia.

Also the 20 ° C temperature is the average value for slow moving streams and is easily duplicated in an incubator. Hence the test conditions for standard BOD test are 20 ° C for 5 days.

Application of BOD data:

1. It is the only test applied that gives a measure of the amount of biologically oxidizable organic matter present that can be used to determine the rates at which oxidization will occur or BOD will be exerted in receiving bodies of water.
2. BOD is therefore a major criterion used in stream pollution control where organic loading must be restricted to maintain desired dissolved oxygen levels.
3. The determination is used in studies to measures the purification capacity of stream and serves regulatory authorities as a means of checking the quality of effluents discharged to such waters.
4. Information concerning the BOD of wastes is an important consideration in the design of treatment facilities.
5. It is a factor in the choice of treatment method and is used to determine the size of certain units, particularly trickling filters and activated sludge units.
6. After treatment plants are placed in operation, the test is used to evaluate the efficiency of process.

Determination of BOD:

The BOD test is based upon determinations of dissolved oxygen before and after a time period of 3 or 5 days. Consequently the accuracy of the results is influenced greatly by the care given to its measurements. There are two methods to measures BOD of sample.
1. Direct Method
2. Indirect Method

Apparatus:

1. Incubation bottles
2. Incubator and water bath
3. Burette
4. Pipette
5. Measuring Cylinder
6. Beaker
7. Conical flask

Reagents:

1. Phosphate buffer solution
2. Magnesium sulphate solution ($MgSO_4$)
3. Calcium chloride solution ($CaCL_2$)
4. Ferric chloride solution ($FeCL_3 . 6H_2O$)
5. Acid and Alkali solution
6. Sodium thiosulphate solution 0.025 N
7. Ammonium chloride solution
8. Manganaous sulphate

1. Direct Method:

Principle:

Direct method is used in case of samples whose 5 days BOD does not exceed 7 mg/lit , it is not necessary to dilute them, provided that they are aerated to bring the dissolved - oxygen level nearly to saturation at the start of the test. Many river waters fall in this category.

Procedure:

1. Adjust the sample to about 20° C and aerate with diffused air to increase the dissolved gas content of the sample to near saturation.
2. Fill two or more BOD bottles with the sample.
3. At least one is analysed for dissolved oxygen immediately.
4. The other bottles are incubated for 5 days at 20° C.
5. After 5 days; the amount of dissolved oxygen remaining in the incubating sample is determined.
6. The 5 days BOD is calculated by subtraction of the 5 day results from those obtained initially.

Significance:

The direct method of measuring BOD involves no modification of the sample, and therefore produces results under conditions as nearly similar possible to the natural environmental.

Unfortunately, the BOD of very few samples falls with the range of dissolved oxygen available in this test.

2. Dilution Method:

Dilution method of measuring BOD is based upon the fundamental concept that the rate of biochemical degradation of organic matter is directly proportional to the amount of unoxidized material existing at the time.

According to this concept, the rate at which oxygen is used in dilutions of the waste is in direct proportion to the percent of waste in the dilution. The major factors of importance are:
1. Freedom from toxic materials.
2. Favourable pH and osmotic conditions.
3. Presence of available accessory nutrient elements.
4. Std. Temperature.
5. Presence of a significant population of mixed organisms of soil origin.

PRETREATMENT REQUIRED:

1.   Dilution of Waste:
In order to ensure that dissolved oxygen will be present throughout the period of the test usually dilution is carried out for all kind of samples or wastes. Usually it is best to set three different dilutions when the strength of a sample is known or else two dilutions may suffice. In any case, there should be an overlapping of the BOD measurable by successive dilution.

It is customary to estimate the BOD of a sample and set 1 dilution based upon the estimate, 2 other dilutions; 1 higher and 1 lower are also set up. In direct-pipetting techniques; preliminary dilutions should be made of all samples that requires less than 0.5 ml of the samples, so that amount added to the bottles can be measured without serious error. The volumes of all bottles must be known in order to allow calculations of the BOD when this method is used.

Dilution Technique:

Dilutions that result in a residual DO of at least 1 mg/lit and a DO uptake of at least 2 mg/lit after 5 day incubation produce the most reliable results. Make several dilutions of prepared samples to obtain DO uptake in this range. Experience with a particular sample will permit use of smaller number of dilutions. A more rapid analysis, such as COD and BOD serve as guide in selecting dilutions. The following dilutions serve as guidelines:

> 0.1 to 1.0 % for strong industrial wastes,

> 1 to5 % for raw and settled wastewater,

> 5 to 25 % for biologically treated effluent, and

> 25 to 100 % for polluted river waters.

Prepare dilutions either in graduated cylinders and then transfer to BOD bottles or prepare directly in BOD bottles. When using graduated cylinder to prepare dilutions, and when seeding is necessary, add seed either directly to dilution water or to individual cylinders before dilution. Seeding of individual cylinders avoids a declining ratio of seed to sample as

increasing dilutions are made. When dilutions are prepared directly in BOD bottles and when seeding is necessary, add seed directly to dilution water or directly to the BOD bottles.

2.   Need for Dilution Water:

A wide variety of waters have been used for BOD analysis.
1. Natural surface waters would appear to be ideal, but they have a number. of disadvantages like variable BOD, variable microorganism population and variable mineral content.
2. Tap water has been used, but it suffers from most of the limitations found in surface water plus the possibility of toxicity form chlorine residuals.
3. Distilled water also cannot for this test since it is devoid of necessary minerals and nutrients required for the growth of micro-organisms.
4. So through long experience, it has been developed that a synthetic dilution water be prepared from distilled or demineralized water by adding sufficient nutrients and saturating it with dissolved oxygen so that most of the variables mentioned above can be kept under control.

Preparation of dilution water:

The quality of the distilled water used for the preparation of dilution water is of importance. It must be free from toxic substances. In many cases it is necessary to dechlorinate the water. The pH of the dilution water may range anywhere from 6.5 to 8.5 without affecting the action of the heterotrophic bacteria. The buffer is essential to maintain favourable pH conditions at all times.
The proper osmotic conditions are maintained by the potassium & sodium phosphates added to provide buffering capacity. In addition, calcium and magnesium salts are added which contribute to the total salt content. Finally, the dilution water should be aerated to saturate it with oxygen before use.

Procedure:

1. Place desired volume of water in a suitable bottle or container.
2. Add 1 ml each of phosphate buffer, $MgSO_4$. $CaCL_2$ and $FeCL_3$ solutions per Liter of water.
3. Seed dilution water, if required.
4. Before use bring dilution water temperature to 20° C.
5. Saturate with DO by shaking in a practically filled bottle or by aerating with aerator or pump.

3.   The Need for Blanks:

At least 3 blanks should be included with each set of BOD samples. BOD is a bioassay test so there is a certain amount of biological variation always involved. Since the blank serves as the reference value from which all calculation of BOD are made, it imparts statistical reliability to some extent. Usually 3 blanks provide such reliability, but each analyst should satisfy particular requirements.

4.   Seed Source:

Population of microorganism capable of oxidizing the biodegradable organic matter in the sample. Domestic waste water unchlorinated or otherwise undisinfected effluents from biological waste treatment plants and surface waters receiving waste water discharges contain satisfactory microbial populations. But some samples like untreated industrial wastes, disinfected wastes, high-temperature wastes or wastes with extreme pH value do not contain a sufficient microbial population. For such wastes it is required to seed the dilution water by adding a population of microorganisms. The preferred seed is effluent from a biological treatment system processing the waste. Where this is not available the supernatant from domestic wastewater after setting at room temperature for at least 1 hour can be used. When effluent from a biological treatment process is used inhibition of nitrification is recommended.

Determine the existence of a satisfactory population by testing the performance of the seed in BOD tests on the sample. BOD values that increases with time of adaptation to a steady high value indicate successful seed adaptation.

5.   Neutralization:

Samples containing alkalinity or acidity must be neutralized to pH 7.0 by using 1 N H_2SO_4 or NaOH.

6.   Dechlorination:

Samples containing residual chlorine compounds are allowed to stand for 1 to 2 hours. The residual chlorine is often dissipated by this method. In case of higher residual chlorine dechlorinate the sample by using sodium sulphite.

7.   Removal of toxicity:

Samples from industries often contain toxic substances or chemicals but presence of such substances will hinder the growth of bacteria and hence samples must be devoid of toxic substances before carrying out the BOD test.

Principle:

The method consists of filling with sample, an airtight bottle of the specified size and incubating it at the specified temperature of 20° C for 5 days. Dissolved oxygen is measured initially and after incubation period of 5 days. BOD is computed from the difference between initial and final DO. Because the initial DO is determined immediately after the dilution is made, all oxygen uptake, including that occurring during the first 15 min. is included in the BOD measurement.

Relevant Standards:

 According to BIS 2490 Part I, the limits for treated BOD effluent in form of 5 days at 20 ^{0}C are-
1. Inland surface water - 30 ppm.
2. Discharge into public water - 350 ppm.

3. On land for irrigation - 100 ppm.
4. Marine disposal - 100 ppm.

Procedure:

1. Prepare dilution water as mentioned above.
2. Add seed if required
3. Decide the dilution factor and number of dilutions required for the particular sample.
4. Accordingly add sample to 1 liter dilution water and fill minimum three bottles for one dilution.
5. The BOD bottles should be filled up in such a way that it forms a water seal at the stopper.
6. Determine initial DO in one of the bottles.
7. Keep remaining two bottles in the incubator for 5 days at 20° C.
8. At the end of period, test it for final DO.
9. Repeat steps 5 to 8 for blank sample also.

Observations data:

Dilution Prepared:

Sample type: _____ Dilution Factor = _____

Number of dilution=_____

Observation Table:

Sr. No.	Sample Description	Percentage dilution	Dilution factor	ml. of N/40 Sodium thiosulphate		
				Initial DO mg / lit	Final DO mg / lit	BOD mg / lit

Calculation:

BOD_5 = [Initial DO of sample - Final DO of sample] - {Initial DO of blank - Final DO of blank]
                                                          Dilution Factor

Result:
BOD of sample _____   =   mg/lit.

.## Discussion of result:

Conclusion:

Quiz:
1. Define the term BOD.
2. Give the significance of BOD.

3. Why is the incubation period of 5 days selected for determining BOD?
4. What is aerobic and anaerobic oxidation of the effluent?
5. Why the BOD values are always less than the COD values.
6. What is dilution water? Why it is used?

Marks: **Signature:**

.

Experiment No: Date:

DETERMINATION OF pH

Aim:

To determine pH value of the given sample.

Theory:

All liquids of which waste is constituents contains free positively charged H+ ions and negatively charged OH⁻ in varying and related proportion. Water itself dissociated to a some extent, a litre of pure water contains one tenth-millionth of gramme (10^{-7} gm) of ionized hydrogen & same hydroxyl ions. If the concentration of OH⁻ ions and H⁺ions is same, the water is said to be neutral water. Thus the pH of the water is nothing but the hydrogen ion concentration.

The pH value is the logarithms, to base 10, of the reciprocal of the hydrogen ion concentration in gram per litre, i.e.

PH = -log (H⁺)
Where, p indicates potential difference between two electrodes and "H" denotes hydrogen ions concentration. The pH of natural water generally lies in the range of 6.0 to 8.5. Most of the natural water are general alkaline due to presence of carbonates. The pH value of the natural water changes due to biological activities, temperature and disposal of industrial waste etc. pH value of waste water is one of the important influencing parameter in treatment of water and waste water. To maintain anaerobic treatment efficiency, the acid producing bacteria and methane producing bacteria must be in state of dynamic equilibrium.

Application of pH data in Environmental Engineering practice:

1. Determination of pH is one of the important objective in biological treatment of the waste water.
2. pH value of range is of immense value for any chemical reaction. A chemical shall be highly effective at a particular pH. Chemical coagulation, disinfection, water softening and corrosion control are governed by pH adjustment.
3. Dewatering of sludge, oxidation of cyanides and reduction of hexavalent chromium in to trivalent chromium also need a favourable pH range.
4. It is used in the calculation of carbonate, bicarbonate, CO_2 corrosion etc.

Environmental Significance:

P^H has no direct adverse effect on health, however a lower value below 4.0 will produce sour tests & higher pH>8.5 a bitter taste. Higher values of pH induces the formation of trithalomethanes which are causing cancer in human being. It also causes scale formation in water heating apparatus and reduces germicidal potential of chloride. pH below 6.5 starts corrosion in pipes, thereby releasing toxic methods such as Zn, Pb, Cd & Cu etc. According to BIS, water for domestic consumption should have pH between 6.5 to 8.5.

Apparatus:

PH meter along with the electrodes,

Beakers,
Glass rod,
Thermometer

Reagents:

PH buffer,
PH strips

(A)   Electrode method-

Principle:

PH is measured by a pH meter using a glass electrode that generates a potential varying linearly with the pH of the solution in which it is immersed.

Procedure:

Warm the pH meter for 10 to 15 min. Measure the temp. of the buffer and set the temp. of buffer with the help of the temp. knob. Dip the electrode into buffer of pH 7. Set the reading of pH 7.00 on display with calibrate knob. Rinse the electrode thoroughly with distilled water and carefully wipe with a tissue paper. Dip the electrode into buffer of pH 4.00 and check the value on display. If reading is other than 4.00, calibrated the instrument to show the reading 4.00 by slop knob. Check the instrument by check key. Rinse the electrode thoroughly with distilled water and carefully wipe with a tissue paper. Calibrate the instrument with standard buffer solutions of pH 9.2. Rinse the electrode thoroughly with distilled water and carefully wipe with a tissue paper. Dip the electrode into the sample solution. Swirl the solution and wait up to the minute for steady reading. The reading is taken after the indicate value remains constant for one minute.

(B)   **Using pH Paper-**

Dip pH paper strip into the solution. Compare the resulting colour of strip with standard colours. This gives approximate value of pH.

(C) Universal Indicator-

Visual comparison method using universal indicator may also be used. Add 2-3 drops of indicator into the sample with standard colour disc for various pH value.

Observation Table:

Sr. No.	Sample Type	pH	
		PH strip	**pH meter**
1	Tap water		

2	Distilled water	
3	Waste water	
4	Unknown	

Result:

1. P^H of tape water
2. P^H of Distilled water
3. P^H of waste water
4. P^H of unknown

Discussion of result:

Conclusion:

Quiz:
1. What is the best pH value of drinking water?
2. What is the ideal pH for drinking water?
3. Why is pH of water 7?
4. Is pH 9.5 water is healthy?
5. What does a pH of 9 mean?

Marks: **Signature:**

Experiment No: Date:

DETERMINATION OF RESIDUAL CHLORINE

Aim:

To determine the concentration of RESIDUAL CHLORINE in the given water sample.

Theory:

Water is required to be disinfected to make it potable. Hypochlorous acid (HOCL) is the most effective disinfectant, which is formed by adding chlorine to water. Chlorine may be applied in gaseous form (CL_2) or in the form of bleaching powder (Ca (OCl_2). Chlorine combines with water to form Hypochlorous and hydrochloric acids.

$$Cl_2 + H_2O \xrightarrow{PH>4} HOCl + HCL$$

$$HOCl \xrightarrow{PH>6} H^+ + OCl^-$$
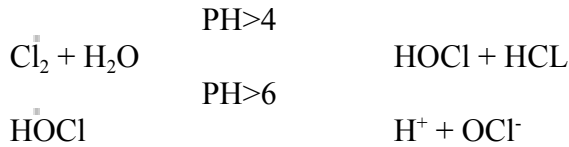
Chlorine and its derivatives (HOCl and OCl) inactivate the enzymatic action of pathogenic bacteria present in water. The sum of Hypochlorous acid (HOCl) & Hypochlorite ions (OCl^-) are called "Free Chlorine Residuals".

Residual chlorine is the chlorine remaining or available in water after the contact time (30 to 60 minutes) in chlorination process. Residual chlorine is required to maintain disinfection and prevent water from the action of pathogenic bacteria during the distribution till it reaches to the consumers. The concentration of residual chlorine should be 0.1 to 0.2 mg/l and chlorine dose required to maintain this concentration is equal to 0.5 to 2.0 mg/l (5.0 to 8.0 mg/l or more for unfiltered river water.

Environmental Significance:

Active chlorine should be determined at each stage in the processing of drinking water and in the water mains in order to guarantee biologically safe water. Active chlorine (residual chlorine) should be present in drinking water within the range of 0.1 to 0.2 mg/l. Excessive chlorination gives bad odour and taste to the drinking waters.

Application of Residual Chlorine data in Environmental Engineering Practice:
1. Residual chlorine determination is used to control chlorination of domestic and industrial waste waters.
2. Determination of residual chlorine is used universally in disinfection practice to control addition of chlorine so as to ensure effective disinfection without waste.
3. Determination of residual chlorine in water distribution is useful to detect the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Determination of Residual Chlorine:

ORTHOTOLIDINE is an aromatic organic compound that is oxidized in acid solution by chlorine, chloramines and other oxidizing agents to produce a yellow coloured compound called as 'Holoquinone'. The intensity of this yellow colour is proportional to the amount of chlorine present.

Apparatus:

Chloroscope.

Reagent:

Orthotolidine reagent.

Procedure:

1. Take the water sample in one of the cylinders of the comparator and distilled water into the other.

2. Add 5 drops of Orthotolidine solution to both the cylinders and put them in the comparator with distilled water cylinder behind the colour scale.
3. The colour which matches in both the cylinders directly gives the 'Residual Chlorine'.

Observation table:

Sr. No.	Sample Type	Residual Chlorine
1		
2		
3		

Result:

Residual Chlorine content of the given water/waste water sample is _____mg/1

Discussion of results:

Conclusion:

Quiz:
1. Discuss the roll of pH in the determination of residual chlorine.
2. What is the action of chlorine on pathogens?
3. Discuss different methods of sterilization of water.
4. Write down the reactions involved in the residual chlorine determination.
5. Name the impurities that interfere in this determination.
6. What is the colour change at the end point and why?

Marks: **Signature:**

Experiment No: Date:

DETERMINATION OF TOTAL DISSOLVED SOLIDS

Aim:

To determine total dissolved solids in the given sample.

Theory:

Total dissolved solids as the name suggests consists of colloidal and dissolved solids having organic and inorganic molecules, which cannot be removed by simple settling. It requires biological oxidation or coagulation followed by sedimentation or reverse osmosis or electro dialysis for its removal. The material that passes through the filter along with the water (called as filter) on evaporation at 104±1°C gives the total dissolved/filterable solids.

Application of Total dissolved solid data in Environmental Engineering Practice:

Estimation of total dissolved solids is useful to determine whether the water is suitable for drinking purpose or industrial purpose.

Principle:

Total dissolved solids are determined as the residue left after evaporation and drying of the filtered sample.

Apparatus:

Oven, Desiccators, what man filter paper No.44, Evaporating dish, Water Bath

Procedure:

1. A Clean porcelain dish is ignited in a muffle furnace and after partial cooling in the air, it is cooled in a desiccators and weighted.
2. A 100 ml of filtered sample is placed in the dish and evaporated at 100 ^{0}C on water bath, followed by drying in oven at 103 ^{0}C for 1 Hour.
3. Dry to a constant weight at 103 ^{0}C, cool in a desiccators and weight.

Observation Table:

Sr. No.	Sample Type	Volume Of Sample	Initial weight of Evaporating dish (W_1)	Final weight of Evaporating dish (W_2)	Difference in weight ($W_2 - W_1$)	Dissolved solids mg/l

Calculations:

Increase in weight (in mg) * 1000

Total Dissolved Solids (mg/ lit) =   --
                                     ml of sample taken

Result:

Total dissolved solids content of the given water/waste water sample is _____mg/1

Discussion of results:

Conclusion:

Quiz:
1. What is the best TDS level for drinking water?
2. Is high TDS value is harmful?
3. How do you calculate total dissolve solids?
4. Does boiling water reduce TDS?
5. What causes high total dissolve solids?
6. What is the difference between TDS and TSS?
7. Are TDS and hardness the same?
8. Is low TDS water is harmful?

Marks: **Signature:**

Experiment No: Date:

CYCLONE SEPARATOR

Objective:

To calculate the efficiency of cyclone separator.

EQUIPMENT AND MATERIAL:

Cyclone Separator, Weighing Scale, brick powder /Sand.

THEORY:

Cyclone separators or simply cyclones are separation devices (dry scrubbers) that use the principle of inertia to remove particulate matter from flue gases.

Cyclone separators is one of many air pollution control devices known as pre cleaners since they generally remove larger pieces of particulate matter.

This prevents finer filtration methods from having to deal with large, more abrasive particles later on.

In addition, several cyclone separators can operate in parallel, and this system is known as a multicyclone.

It is important to note that cyclones can vary drastically in their size. The size of the cyclone depends largely on how much flue gas must be filtered, thus larger operations tend to need larger cyclones.

For example, several different models of one cyclone type can exist, and the sizes can range from a relatively small 1.2-1.5 meters tall (about 4-5 feet) to around 9 meters (30 feet)—which is about as tall as a three story building!.
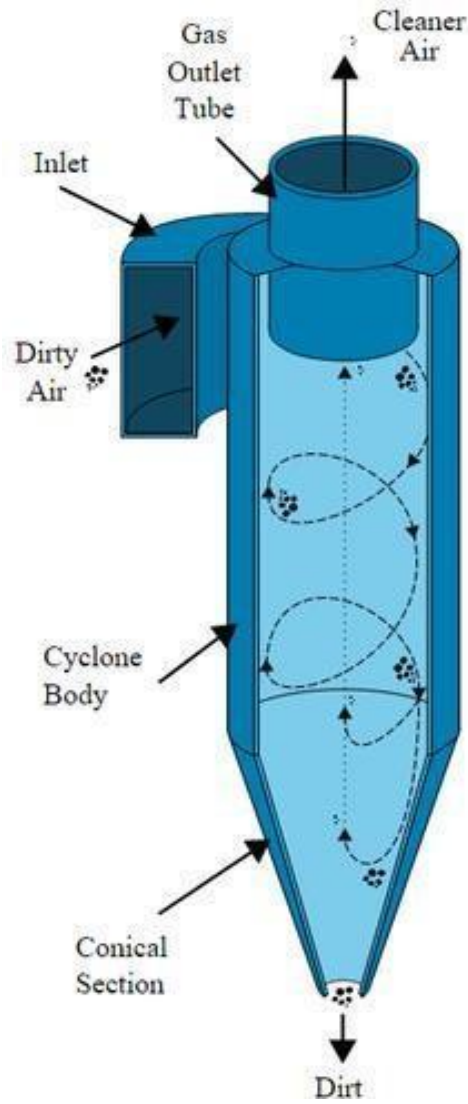
PROCEDURE:

Weigh_____gm of the sample. (brick powder /Sand)

Fill the sample in the hopper of the cyclone.

Switch on the blower with 100% opening of the air valve and determine the air velocity with the help of anemometer.

Measure the amount of sample collected in the bottom jar/flask.

Repeat the above procedure with different flow rate of air and find out the efficiency.

FIG: CYCLONE SEPARATOR

Observation:

1. Initial weight of feed taken   =_____ gms.

2. Weight of material collected =_____ gms.

Calculation:

Efficiency of cyclone when 100% valve is

open η = <u>Amount Collected</u>   X

100

Amount fed

Result:

Efficiency of Cyclone:-

CONCLUSION:

Quiz:-

1. What is cut point diameter (Dpc) of Cyclone Separator.
2. Why Cyclone Separator is used in series?
3. Explain industrial application of Cyclone Separator.

Marks: **Signature:**

Experiment No: Date:

FREUANDLICH
ADSORPTION ISOTHERM

Objective:

To plot Freundlich adsorption isotherm for a system of oxalic acid and activated charcoal.

Apparatus:

Conical flask, filtration unit, measuring cylinder, weight balance, physical balance

Chemicals:

Oxalic acid (1N), charcoal .NAOH

Procedure:

- Prepare 1N oxalic acid sol^n. Add 10, 20,30,40,50, ml of this sol^n in conical flask. No read respectively.

- Add 90, 80,70,60,50 ml of water in conical flask or no read 1, 2,3,4,5, respectively.

- Add 5gm of charcoal in each flask.

- Mix the sol^n well and after 30 min. Filter the sol^n

- Adjust the no. Of suitable flask required after filtration. Titrate 10 ml filtrate with 0.1 N NAOH sol^n and find out normality.

- Draw the graph ln C $\longrightarrow$ ln x values.

Freundlich equation curves:-

OBSERVATION:-

- Burette:-.......................NAOH sol^n
- Pipette:-....x n...............oxalic acid sol^n
- Indicator:- phenolphthalein
- End product:- colorless to pink
- Mass of charcoal m: -.............

OBSERVATION TABLE:-

SR. NO	1	2	3	4	5
VOL^M OF OXALIC ACID IN FLASK(ML)					
VOL^M OF H$_2$O IN FLASK(ML)					
VOL^M OF FILTRATE (ML)					
VOL^M OF FILTRATE FOR TITRATION(ML)					
BURETTE READING					
NORMALITY OF FILTRATE					
LN C					
WT. OF ACID ADSORBED X(GM)					
X= x/m					
LN X					

CALCULATION [FOR STEP 1]:-

1) **Normality of oxalic acid:-**

N_1V_1 (NAOH) = N_2V_2 (OXALIC ACID)

N_2 =................

C =..................

2) GM OF ACID FILTERED (X) = N × EQ. WT. × VOLM OF FILTRATE

$X=$

$X=$......................GM

3) X = x/m

$X =$

$X=$

4) LN X = LN (   )

LN X =

Similarly the other next four readings to be calculated.

RESULT:-

- Value of slope of Freundlich isotherm m =**.....................**
- Value of k-line   **n =................**

CONCLUSION:-

Quiz:
1. What do you understand by the term adsorption?
2. Distinguish between adsorption and absorption?
3. What is the effect of temperature on adsorption of gases on solids?
4. What are adsorption isotherms? What information can be obtained from them?
5. Distinguish between physical adsorption and chemical adsorption.

Marks: **Signature**