Channabasaveshwara Institute of Technology

(An ISO 9001:2015 Certified Institution)

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Department of Civil Engineering

Environmental Engineering Laboratory

(18CVL67)

B.E – VI Semester

Name : _____

USN : _____

Batch : _____ Section : _____

List of Abbreviations

BOD	Biochemical Oxygen Demand		nm	Nanometer
BP	Bleaching Powder		NTU	Nephelometric Turbidity unit
BR	Burette Reading		N	Normality
COD	Chemical Oxygen Demand		NBP	Normality of bleaching
DO	Dissolved Oxygen		PAH	Polynuclear Aromatic Hydrocarbons
DW	Distilled Water		Qty	Quantity
DS	Double Strength		rpm	revolutions per minute
EDTA	Ethylene Diamine Tetraacetic Acid		S	Siemen
FAS	Ferrous Ammonium Sulphate		SS	Single Strength
LPG	Liquefied Petroleum Gas		TDS	Total Dissolved Solids
MBAS	Methylene Blue Active Substances		UV-	UltraViolet- Visible
μg	Microgram		VBP	Volume of bleaching powder
ml	milli liter		Wt	Weight
mS	milli Siemen			
mg	Milligrams			
μS	micro Siemen			
mg/L	milligrams per Liter			
M	Molarity			
MPN	Most Probable Number			

Chemical Formulae

AgNO3	Silver nitrate		CH3COOH	Acetic acid
K2CrO4	Potassium chromate		NH4C2H3O2	Ammonium acetate
NaCl	Sodium Chloride		C12H8N2.H2O	Phenanthroline monohydrate
Cl	Chloride		KI	Potassium iodide
H2SO4	Sulfuric acid		NH2OH.HCl	Hydroxyl amine
NaOH	Sodium hydroxide		NaC2H3O2.H2O	Sodium acetate
CaSO4	Calcium sulphate		ZrOCl2.8H2O	Zirconyl chloride octahydrate
MgSO4	Magnesium sulphate		Na	Sodium
HCl	Hydrochloric acid		K	Potassium
Na2SO4	Sodium sulfate		HNO3	Nitric acid
CO2	Carbon dioxide		NO3	Nitrate
Na2S2O3	Sodiumthiosulphate			
CaCO3	Calcium carbonate			
MnSO4.XH2O	Manganese sulphate			
MnO2	Manganese oxide			
H2O	Water			
Mn(OH)2	Manganese hydrooxide			
CaCl2	Calcium chloride			
FeCl3	Ferric chloride			
K2Cr2O7	Potassium dichromate			
Ag2SO4	Silversulphate			
Fe (NH4)2 SO4	Ferrous ammonium sulphate			

Bureau of Indian Standards Specification for Drinking Water

(BIS 10500-1991)

Substance or characteristic	Requirable (Desirable Limit)	Undesirable effect outside the Desirable Limit	Permissible Limit in absence of Alternate source
Colour, Hazen	05	Above 5,consumer acceptance decreases	25
Odour	Unobjectionable		
Taste	Agreeable		
Turbidity, NTU,	5	Above 5, consumer acceptance decreases	10
pH value	6.5 - 8.5	Beyond this range the water will affect the mucous membrane and / or water supply system	No relaxation
Total hardness(as CaCO3) mg/L	300	Encrustation in water supply structure and adverse affect on domestic uses	600
Iron (as Fe) mg/L, max	0.3	Beyond this limit taste / appearances are affected, has adverse affect on domestic uses and water supply structures, and promotes iron Bacteria	1.0
Chlorides (as Cl) mg/L	250	Beyond this limit, taste, corrosion and palatability are affected	1000
Residual, free chlorine, mg/L,	0.2		
Dissolved solids mg/L, max	500	Beyond this palatability decreases and may cause gastro intestinal irritation	2000
Calcium (as Ca) mg/L, max	75	Encrustation in water supply structure and adverse effects on Domestic use	200
Copper (as Cu) mg/L, max	0.05	Astrigent taste, discolouration and corrosion of pipes, fittings and utensils will be caused beyond this	1.5
Manganese as Mn mg/L, max	0.1	Beyond this limit taste /appearances are affected, has adverse affect on domestic uses and water supply structures	0.3
Sulphate (as SO4) mg/L, max	45	Beyond this Methaemoglobinemia takes place in infants	100
Fluoride, (as F) mg/L, max	1.0	Fluoride may be kept as low as possible. High fluoride may cause fluorosis	1.5
Phenolic compounds (as C6H5OH) mg/L, max	0.001	Beyond this, it may cause objectionable taste and odor	0.002
Mercury (as Hg) mg/L, max	0.001	Beyond this, the water becomes toxic	No relaxation

Substance or characteristic	Requirable (Desirable Limit)	Undesirable effect outside the Desirable Limit	Permissible Limit in absence of Alternate source
Cadmium (as Cd) mg/L, max	0.01	Beyond this, the water becomes toxic	No relaxation
Selenium (as Se) mg/L, max	0.01	Beyond this, the water becomes toxic	No relaxation
Arsenic (as As) mg/L, max	0.05	Beyond this, the water becomes toxic	No relaxation
Cyanide (as CN),mg/L,max	0.05	Beyond this, the water becomes toxic	No relaxation
Lead (as Pb) mg/L, max	0.05	Beyond this, the water becomes toxic	No relaxation
Zinc (as Zn) mg/L, max	05	Beyond this limit it can cause astringent taste and an opalescence in water	15
Anionic detergents (as MBAS) mg/L, max	0.2	Beyond this limit it can cause a light froth in water	1.0
Chromium (as 0.05 Cr^{6+}) mg/L, max	0.05	May be carcinogenic above this limit	No relaxation
Polynuclear aromatic hydrocarbons (as PAH) mg/L, max		May be carcinogenic	
Mineral oil, mg/L, max	0.01	Beyond this limit undesirable taste and odour after chlorination takes place.	0.03
Pesticides, mg/L,max	Absent	Toxic	0.001
Radioactive materials:			
1. Alpha emitters Bq/L, max			
2. Beta emitters pci/L, max			
3. Alkalinity, mg/L, max	200	Beyond this limit taste becomes unpleasant	600
4. Aluminium, (as Al), mg/L, max	0.03	Cumulative effect is reported to cause dementia	0.2
5. Boron, (as Bo), mg/L, max	01		05
6. Magnesium, (as Mg), mg/L, max	30	Encrustation to water supply structure and adverse effects on domestic use.	100

Treated Effluent Standards General Standards for Discharge of Environmental Pollutants: Sewage
(As Per CPCB, New Delhi)

Parameter	STANDARDS			
	Inland surface water	Public sewers	Land for irrigation	Marine coastal areas
Suspended solids mg/L, max.	100	600	200	• For process waste water 100 • For cooling water effluent 10% above total suspended matter of effluent
Particle sizes of Suspended solids	Shall pass 850 micron IS sieve			• Floatable solids, Max. 3mm size • Settelable solids,max. 850 microns
pH value	5.5 - 9.0	5.5 - 9.0	5.5 - 9.0	5.5 - 9.0
Temperature	Shall not exceed 05^0 C above the receiving water Temperature			Shall not exceed 5^0C above the receiving water temperature
Oil and grease, mg/L, max	10	20	10	20
Total residual chlorine, mg/L, max	1.0	-	-	1.0
Ammonical nitrogen (as N), mg/L, max.	50	50	-	50
Total kjeldhal nitrogen [N], mg/L, max	100	-	-	100
Color and odor	*	-	*	*
Free ammonia as (NH3) mg/L, max	5.0	-	-	5.0
Biochemical Oxygen Demand (5 day at 20^0), mg/L max. (BOD5)	30	350	100	100
Chemical Oxygen Demand (COD)	250	-	-	250
Arsenic (as As), mg/L, max	0.2	0.2	0.2	0.2
Mercury (as Hg) mg/L, max	0.01	0.01	-	0.01
Lead (as Pb) mg/L, max	0.1	1.0	-	2.0

Parameter	STANDARDS			
	Inland surface water	Public sewers	Land for irrigation	Marine coastal areas
Calcium (as Ca) mg/L, max	2.0	1.0	-	2.0
Hexavalent Chromium (as Cr) mg/L, max	0.1	2.0	-	1.0
Total Chromium (as Cr) mg/L,max	2.0	2.0	-	2.0
Copper (as Cu) mg/L, max	3.0	3.0	-	3.0
Zinc (as Zn) mg/L, max	5.0	15	-	15
Selenium (as Se) mg/L, max	0.05	0.05	-	0.05
Nickel (as Ni) mg/L, max	3.0	3.0	-	5.0
Cyanide (as CN) mg/L, max	0.2	2.0	0.2	0.2
Fluoride (as F) mg/L, max	2.0	15	-	15
Dissolved Phosphate (as P) mg/L, max	5.0	-	-	-
Sulphide (as S) mg/L, max	2.0	-	-	5.0
Manganese (as Mn) mg/L	02	02	-	02
Iron (as Fe) mg/L	03	03	-	03
Vanadium (as V) mg/L	0.2	0.2	-	0.2
Nitrate Nitrogen mg/L	10	-	-	20
Radioactive materials, 1. Alpha emitters [micron curie/mL] max	10^{-7}	10^{-7}	10^{-8}	10^{-7}
2. Beta emitters [micro curie /mL] max	10^{-6}	10^{-6}	10^{-7}	10^{-6}

NOTE: * = All efforts should be made to remove color and odor as far as practicable.

Introduction

1. Do not add water to concentrated acid. A concentrated acid should be added carefully to water.
2. Do not handle chemicals with bare hands.
3. Do not blowout the last drop from the pipette when the liquid is drained out completely. Touch the tip of pipette to the inner surface of the vessel.
4. Read the level of the curve (Miniscus) in all volumetric glass coarse with the eye approximately the same level as the curve of the solution.
5. Clean all the apparatus used with distilled water before and after the experiment.

NORMALITY (N): The number of gram equivalent weight of substance dissolved in 1 liter of water is called Normality.

For Example: The gram equivalent weight of HCl is 36.5 grams. So, if 36.5 grams of HCl is added to 1 liter of water, it gives a solution of 1N.

Similarly for oxalic acid = 90 / 2 = 45 grams in 1 liter of water, it gives a solution of 1N.

MOLARITY (M): The number of gram molecular weight of the substance dissolved in 1 liter of water is called Molarity.

For Example: The gram molecular weight of oxalic acid is 90 grams. If 90 grams of Oxalic acid is dissolved in 1 liter of water it is called 1 molar solution. (1M)

MOLALITY: The number of gram moles of substance dissolved in 1 Kg (1000 grams) of the solvent.

For Example: If 40 grams Of NaOH (Molecular weight = 40) of Sodium hydroxide is dissolved in 1 Kg of water, the molarity of the solution is 1.

For water: **mg/L = ppm**

Tabulation

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF H2SO4 USED
			FR	IR	FR-IR	
1.	Water Sample supplied in lab	Phenolphthalein				
2.						
3.						
4.		Methyl Orange				
5.						
6.						

Calculation

A. Phenolphthalein alkalinity as $CaCO_3$ = $\dfrac{V_1 \times N \times 50 \times 1000(mg/l)}{ml\ of\ sample\ used}$

B. Methyl Orange Alkalinity as $CaCO_3$ = $\dfrac{V_2 \times N \times 50 \times 1000(mg/l)}{ml\ of\ sample\ used}$

C. Total Alkalinity as $CaCO_3$ = Phenolphthalein alkalinity as $CaCO_3$ + Methyl Orange alkalinity as $CaCO_3$

. **Alkalinity Relationship (P and T)**

➢ Results of titration =

➢ ☐Hydroxide Alkalinity/Caustic Alkalinity as $CaCO_3$ =

➢ Carbonate Alkalinity as $CaCO_3$ =

➢ ☐Bicarbonate Alkalinity as HCO_3 =

Expt. No. 1   **Date:** _____

<u>ALKALINITY TEST</u>

Aim of the Experiment:

To determine the Phenolphthalein Alkalinity and Methyl orange Alkalinity or Total Alkalinity of a given sample of water.

Methodology:

The sample is titrated against Standard Acid using Phenolphthalein and Methyl orange indicator (using titration).

Apparatus:

1. 250 ml cap. Conical flask

2. 250 ml cap. Beaker

3. 50 ml Burette

4. 25 ml pipette

Reagents Used:

1. Standard Sulphuric Acid of 0.02 N

2. Phenolphthalein indicator

3. Methyl orange indicator

4. 0.1 N Sodium Thiosulphate

5. CO_2 Free Distilled Water

Theory:

Alkalinity of water is its quantitative capacity to neutralize a strong acid to a designated pH. Alkalinity is significant in many uses and in treatment of natural acid and wastewater. It is significant in determining suitability of water for drinking as well as irrigation purpose. Alkalinity measurements are used in interpretation and control of water and wastewater treatment process. Alkalinity is primarily due to salts of weak acids and bicarbonates. Major part of alkalinity is because of the action of Carbon dioxide on basic materials.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Phenolphthalein alkalinity as $CaCO_3$	_____ mg/l
2.	Methyl Orange alkalinity as $CaCO_3$	_____ mg/l
3.	Total alkalinity as $CaCO_3$	_____ mg/l
4.	Hydroxide alkalinity as $CaCO_3$	_____ mg/l
5.	Carbonate alkalinity as $CaCO_3$	_____ mg/l
6.	Bicarbonate alkalinity as $CaCO_3$	_____ mg/l

Comments:

Procedure:

A. Phenolphthalein Alkalinity

1. Pipette out 25 ml of given sample of water into a 250 ml conical flask.
2. Add 1 drop of 0.1 N Sodium Thiosulphate solution to remove free residual chlorine, if present.
3. Add 2 to 3 drops of phenolphthalein indicator.
4. If the sample turns pink in color, then titrate with 0.02N standard H_2SO_4, till the pink color just disappears.
5. The end point is when the pink color changes to colourless.
6. Note down the volume of acid is used (V_1).
7. If the pink color does not exist it indicates that, Phenolphthalein Alkalinity is absent. Then continue the titration with Methyl orange indicator.

D. Methyl Orange Alkalinity
1. Add 2 to 3 drops of Methyl orange indicator to the sample.
2. If the solution turns yellow, continue the titration with 0.02N standard H2SO4till the solution turns to faint orange color.
3. Note down the total volume of acid used (V2).

Observation:

1. Conical Flask   :   25 ml of water sample

2. Burette   :   0.02N Sulphuric Acid (H_2SO_4)

3. Indicators   :   Phenolphthalein and Methyl orange

4. End point   :   a) Pink to colorless

                            b) Yellow to Faint orange

Alkalinity Relationship (P and T)

The values obtained from Phenolphthalein and Total alkalinity determination for a given sample of water are used to estimate three forms of alkalinity shown below.

Results of Titration	Hydroxide Alkalinity/Caustic Alkalinity asCaCO$_3$	Carbonate Alkalinity as CaCO$_3$	Bicarbonate Alkalinity as CaCO$_3$
P=0	0	0	0
P< ½ T	0	2P	T – 2P
P= ½ T	0	2P	0
P> ½ T	2P – T	2(T - P)	0
P= T	T	0	0

Where, P = Phenolphthalein alkalinity

T = Total alkalinity

Observation:

Conical Flask	:	25 ml of water sample
Burette	:	0.02N NaOH
Indicators	:	Phenolphthalein and Methyl orange
End point	:	a) Yellow to Faint orange
		b) Colorless to faint pink color

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF NaOH USED
			FR	IR	FR-IR	
1.						
2.	Water Sample supplied in lab	Phenolphthalein				
3.						
4.						
5.		Methyl Orange				
6.						

Calculation

A. Methyl Orange acidity as $CaCO_3$ = $\dfrac{V_1 \times N \times 50 \times 1000}{\text{ml of sample used}}$ (mg/l)

B. Phenolphthalein acidity or Total acidity as $CaCO_3$ = $\dfrac{V_2 \times N \times 50 \times 1000}{\text{ml of sample used}}$ (mg/l)

Expt. No. 2   **Date:** _____

ACIDITY TEST

Aim of the Experiment:

To determine the Methyl orange Acidity and Phenolphthalein Acidity or Total Acidity of a given sample of water.

Methodology:

The sample is titrated against Standard alkaline reagent using phenolphthalein and Methyl orange indicator (using titration).

Apparatus:

1. 250 ml cap. Conical flask
2. 250 ml cap. Beaker
3. 50 ml Burette
4. 25 ml pipette

Reagents Used:

1. Standard NaOH of 0.02 N
2. Phenolphthalein indicator
3. Methyl orange indicator
4. 0.1 N Sodium Thiosulphate
5. CO_2 Free Distilled Water

Theory:

Acidity of water is its quantitative capacity to neutralize a strong base to a designated pH. Acidity of water is significant in many water supply systems, because acidity influences certain chemical and biological process in water.

Acidity of water is due to Hydrogen ions (H^+) present in a sample of water. As a result, dissociation of Hydrogen ions is neutralized by titration with standard solution of a strong base in presence of an indicator. Dissolved Carbon dioxide is usually the major acidic component of surface water. Acidity is usually determined by titration with 0.02N solution of Sodium Hydroxide. The amount of Sodium Hydroxide required for the sample to reach the pH 4.5 in a measure of Methyl orange acidity and amount to reach pH 8.3 is a measure of total acidity.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Methyl Orange acidity as CaCO3	_____ mg/l
2.	Phenolphthalein acidity as CaCO3 Or Total acidity as CaCO3	_____ mg/l

Discussion/Comments on Results:

Procedure:

A. Methyl Orange Acidity

1. Pipette out 25 ml of given sample of water into a 250 ml conical flask.

2. Add 1 drop of 0.1 N Sodium Thiosulphate solution to remove free residual chlorine, if present.

3. Add 2 to 3 drops of methyl orange indicator to the sample. If solution turns yellow, note down the pH value and stop the experiment. If the solution changes to faint red color, titrate against 0.02N NaOH solution upto faint orange color.

4. Note down the volume of titrant used (V_1)

B. Phenolphthalein Acidity

1. Pipette out 25 ml of given sample of water into a 250 ml conical flask.

2. Add 1 drop of 0.1 N Sodium Thiosulphate solution to remove free residual chlorine, if present.

3. Add 2 to 3 drops of phenolphthalein indicator.

4. If the sample turns pinks color it means that, phenolphthalein acidity is absent and stop the experiment.

5. If there is no color change, the titrate with 0.02N Standard NaOH solution till faint pink color appears. The end point is colorless to faint pink color.

6. Note down the volume of titrant used (V_2).

Observation:

Conical Flask	:	25 ml of water sample
Burette	:	Standard EDTA solution of 0.01M
Indicators	:	Erichrome Black – T
End point	:	Wine Red to Blue

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF EDTA USED
			FR	IR	FR-IR	
1.	Water Sample supplied in lab	Erichrome Black – T				
2.						
3.						
4.	Boiled and cooled water					
5.						
6.						

Calculation:

1. Total Hardness as $CaCO_3$ in mg/l = $\dfrac{V_1 \times B \times 1000}{\text{ml of sample used}}$

2. Permanent Hardness as $CaCO_3$ in mg/l = $\dfrac{V_2 \times B \times 1000}{\text{ml of sample used}}$

   Where, B=1 i.e., 1 ml of EDTA solution = 1 mg of $CaCO_3$ = 0.01M EDTA

3. Temporary Hardness as $CaCO_3$ in mg/l= (Total hardness–Permanent hardness)

Expt. No. 3   **Date:** _____

HARDNESS TEST

Aim of the Experiment:

To determine the Total Hardness, Carbonate Hardness and Non-Carbonate Hardness of a given sample of water.

Methodology:

EDTA, Titrimetric method.

Apparatus:

1. 250 ml cap. Conical flask
2. 250 ml cap. Beaker
3. 50 ml Burette
4. 25 ml pipette

Reagents used:

1. Standard EDTA solution of 0.01M (Ethylene Diamine Tetra Acetic Acid).
2. Ammonia Buffer solution.
3. Erichrome Black – T indicator (EBT).

Theory: Originally hardness of water is a measure of soap consuming capacity to produce foam or lather. Also produce scale in hot water pipes, heaters, boilers and other units, precipitated chiefly by Calcium and Magnesium ions commonly present in water. Also may be precipitated by ions of other poly– valent metals such as Aluminium, Iron, Strontium, Zinc and by Hydrogen ions. Calcium and Magnesium ions are usually present in significant concentrations in natural water. Hardness of water varies from place to place. Surface waters are soften than the ground water, due to the dissolved salts of Carbonates, Bicarbonates, Chlorides, Sulphate, Nitrates of Calcium and Magnesium. Temporary hardness is mainly due to Carbonate and Bicarbonate of Calcium and Magnesium. Permanent hardness is mainly due to Sulphateand Chlorides. The hardness of water is expressed in $CaCO_3$ in mg/l.EDTA compound or Sodium salts are soluble complex of a certain metallic cation.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Total Hardness as $CaCO_3$	_____ mg/l
2.	Permanent Hardness as $CaCO_3$	_____ mg/l
3.	Temporary Hardness as$CaCO_3$	_____ mg/l

Discussion/Comments on Results:

A small amount of dye Erichrome Black –T is added to hard water containing Calcium and Magnesium ion, the solution will become wine red. EDTA is then added as a titrant. Complexes of Calcium and Magnesium are formed after sufficient EDTA has been added. The solution turns from Wine Red to Blue. This is the end point of titration, a pH value of 10.2 is usually maintained.

Procedure:

A. Total Hardness

1. Pipette out 25 ml of given sample of water into a 250 ml conical flask.
2. Add 0.5ml of Ammonia Buffer solution to maintain a pH of 10 ± 0.2 and mix well.
3. Add a pinch of Eriochrome Black – T indicator powder. The color of the solution turns Wine Red.
4. Titrate immediately with Standard EDTA solution by slowly mixing until the color changes from Wine Red to Blue.
5. Note down the ml of titrant used (V_1).

B. Permanent Hardness

Take 100ml of water sample, boil and cool, then take 25ml of water from this, in a conical flask. Follow the same procedure as in total hardness. Note down the volume of EDTA used (V_2).

C. Temporary Hardness

Temporary hardness is mainly due to Carbonate and Bicarbonate of Calcium and Magnesium. This can be determined using Total and Permanent hardness values and is expressed as $CaCO_3$ in mg/l.

Temporary Hardness as $CaCO_3$ in mg/l= (Total hardness–Permanent hardness)

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF EDTA USED
			FR	IR	FR-IR	
1.		Erichrome Black – T				
2.	Water Sample supplied in lab					
3.						
4.						
5.		Murixide indicator				
6.						

Calculation:

A. Total Hardness as $CaCO_3$ in mg/l = $\dfrac{V_1 \times B \times 1000}{\text{ml of sample used}}$

B. Calcium Hardness as $CaCO_3$ in mg/l = $\dfrac{V_2 \times B \times 1000}{\text{ml of sample used}}$

where, B=1 i.e., 1 ml of EDTA solution = 1 mg of $CaCO_3$ = 0.01M EDTA

C. Calcium Hardness as Ca in mg/l = (0.40 x Calcium Hardness as $CaCO_3$)

D. Magnesium Hardness as $CaCO_3$ in mg/l
       = (Total hardness as $CaCO_3$- Calcium Hardness as $CaCO_3$)

E. Magnesium Hardness as Ca in mg/l
       = (0.24 x Magnesium hardness as $CaCO_3$)

Expt. No. 4   **Date:** _____

CALCIUM AND MAGNESIUM TEST

Aim of the Experiment:

To determine the Calcium hardness and Magnesium hardness of a given sample of water.

Methodology:

Titration Method.

Apparatus:

1. 250 ml cap. Conical flask
2. 50 ml Burette
3. 25 ml pipette

Reagents Used:

1. Standard Sodium Hydroxide solution of 2N.
2. Murexide Indicator.
3. Standard EDTA solution of 0.01M.

Theory:

Calcium and Magnesium are common constituents of natural water impurities contributing to hardness in water. Calcium and Magnesium content may range from zero to several hundreds of milligrams per liter depending on source of water. These salts breakdown in heating to form harmful scale in boilers, pipes and cooking utensils. Small concentration of Calcium Carbonate forms corrosion in metallic pipes by scale formation. Softening treatment methods like Reverse Osmosis, Electro Dialysis, Ion Exchange etc., are used to reduce the levels of Calcium and Magnesium in water required for potable and industrial purpose.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Total Hardness as $CaCO_3$	_____ mg/l
2.	Calcium Hardness as $CaCO_3$	_____ mg/l
3.	Calcium Hardness as Ca	_____ mg/l
4.	Magnesium Hardness as $CaCO_3$	_____ mg/l
5.	Magnesium Hardness as Mg	_____ mg/l

Discussion/Comments on Results:

Procedure:

Part – A (Total Hardness)

1. Pipette out 25 ml of given sample into a 250 ml conical flask.

2. Add 0.5 ml of Ammonia Buffer Solution to maintain a pH 10 ± 0.2 and mix well.
3. Add a pinch of Erichrome Black – T indicator powder. The color of the solution turns to Wine red.

4. Titrate immediately with standard EDTA solution by slowly mixing until the color changes from Wine red to blue.
5. Note down the ml of titrant used (V_1).

Part – B (Calcium Analysis)

1. Pipette out 25 ml of given sample into a 250 ml conical flask.

2. Add 1ml of NaOH solution of 2N to raise pH value to about 12.

3. Add a pinch of Murexide indicator powder and color changes to pink.

4. Titrate immediately with EDTA solution till pink color changes to purple and note down the volume of EDTA used (V_2).
5. Run a reagent blank and keep it aside to complete the end point of sample titrations.

Observation:

1. Conical Flask   :   a) 25 ml of water sample

                                            b) 50 ml of distilled water

2. Burette   :   Standard $AgNO_3$ solution of 0.0141N

3. Indicators   :   Potassium Chromate solution

4. End point   :   Yellowish green to reddish brown

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF $AgNO_3$ USED
			FR	IR	FR-IR	
1.	Water Sample supplied in lab	Potassium Chromate				
2.						
3.						
4.	Blank test distilled water					
5.						
6.						

Calculation:

Chloride as Cl in mg/l = $\dfrac{(V_1 - V_2) \times N \times 35.45 \times 1000}{\text{ml of sample used}}$

Expt. No. 5   **Date:** _____

CHLORIDE TEST

Aim of the Experiment:

To determine the amount of Chloride in a given sample of water.

Methodology:

Mohr's method (Argentometric), the sample after neutralization is titrated with Silver Nitrate solution.

Apparatus:

1. 250 ml cap. Conical flask
2. 250 ml cap. Beaker
3. 25 ml pipette
4. 50 ml Burette

Reagents Used:

1. Chloride free distilled water.
2. Potassium Chromate (K_2CrO_4) indicator.
3. Standard Silver Nitrate ($AgNO_3$) solution of 0.0141 N.

Theory:

Chloride in the form of Chlorine ion is one of the major inorganic anions. The salty taste produced by Chloride concentration is variable and dependent on the chemical composition of water. The Chloride is higher in wastewater than in raw water. Along the sea costal, Chloride may be present in high concentrations because of intrusion of salt water into the water and sewage system.

In this method, slightly alkaline solution is used. Potassium chromate can indicate the end point of Silver nitrate titration of Chloride as quantitatively precipitated before red Silver chromate is formed.
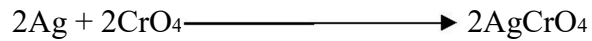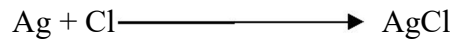
$$NaCl + AgNO_3 \longrightarrow NaNO_3 + AgCl$$
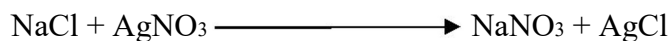
Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Chloride in water, as Cl	_____ mg/l

Discussion/Comments on Results:

In this titration, Chloride ions form white precipitate

$$Ag + Cl \longrightarrow AgCl$$

$$2Ag + 2CrO_4 \longrightarrow 2AgCrO_4$$

The pH must be in the range of 7 to 8 because Ag^+ is precipitated as Silver Hydroxide (AgOH) at high pH.

Procedure:

Part – A (Sample Test)

1. Pipette out 25 ml of given sample into a 250 ml conical flask.

2. Adjust the pH using dilute acid or dilute alkali solution.

3. Add 2 to 3 drops of Potassium Chromate indicator. The color of the solution turns to yellowish green.
4. Titrate against standard $AgNO_3$ solution of 0.0141N.

5. End point is yellowish green to reddish brown color or brick red color.

6. Note down the volume of $AgNO_3$ used as V_1.

Part – B (Blank Test)

1. Take 25 ml distilled water in a conical flask and repeat the procedure from steps in Part A from 2 to 5.
2. The blank showing the end points color should be placed near the sample being titrate to acid in detection of color change and note down the volume of titrant used (V_2).

Observation:

Conical Flask	:	200 ml of chlorinated water sample
Burette	:	Standard Sodium thiosulphate of 0.01N
Indicators	:	Starch solution
End point	:	Blue to colorless

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF Na$_2$S$_2$O$_3$USED
			FR	IR	FR-IR	
1.	Chlorinated water supplied in	Starch solution				
2.						
3.						

Calculations:

Residual Chlorine as Chlorine in mg/l = $\dfrac{\text{V x N x 35.45 x 1000}}{\text{ml of sample used}}$

Expt. No. 6   **Date:** _____

<u>RESIDUAL CHLORINE TEST</u>

Aim of the Experiment:

Estimation of Residual Chlorine in a given sample of water.

Methodology:

Iodometric method

Apparatus:

1. 500 ml cap. Conical flask
2. 100 ml cap.Measuring jar
3. 25 ml pipette
4. 50 ml Burette

Reagents Used:

1.Standard Sodium thiosulphate solution of 0.01 N.
2. Potassium Iodide (KI) crystals.
3. Glacial Acetic Acid.
4. Starch indicator Solution.]

Theory:

Since chlorine is the widely employed method for disinfection the presence of chlorine is common in potable water where chlorinated industrial effluents and sewage are discharged.The primary function of chlorination in water and wastewater is to destroy the disease causing organisms and the overall improvement of water quality.

Chlorine in water may be present as free available chlorine or hypochlorite ion or both and as combined chloride. Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form combined available chlorine. Both free available chlorine and combined available chlorine liberates free Iodine with Potassium Iodide. The liberated Iodine is titrated with standard Sodium thiosulphate solution using starch as an indicator.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Residual Chlorine as Chlorine	_____ mg/l

Discussion/Comments on Results:

Procedure:

1. Take 200ml of chlorinated water sample in a conical flask.

2. Add 5ml of Acetic acid and mix well. Note down the pH value, it should be 3 to 4.
3. Add 1gram of Potassium Iodide crystals and mix well.

4. Titrate immediately with Sodium thiosulphate solution (0.01 N) till light yellow color appears.
5. Add 1ml of Starch indicator, the yellow color changes to dark blue color.

6. Continue the titration till the blue color just disappears. Note down the volume of titrant used (V)

The reaction is preferably carried out in a pH of about 3 to 4

$$2KI + Cl_2 \longrightarrow 2KCl + I_2$$

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaCl + 2NaI$$
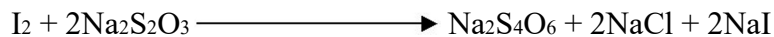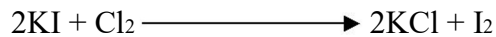
Observation:

1. Conical Flask   :   25 ml of Bleaching powder solution

2. Burette   :   Standard Sodium thiosulphate of 0.025N

3. Indicators   :   Starch solution

4. End point   :   Blue to colorless

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF Na$_2$S$_2$O$_3$USED
			FR	IR	FR-IR	
1.	Bleaching powder solution	Starch solution				
2.						
3.						

Calculations:

mg of Chlorine present in 1mg of Bleaching powder = $\underline{V x\ N\ x\ 35.45}$

ml of sample used

Therefore 1mg of bleaching powder contains X mg of Chlorine

Percentage of Chlorine in Bleaching powder =

Expt. No. 7   **Date:** _____

CHLORINE TEST

Aim of the Experiment:

Estimation of percentage of Chlorine available in a given sample of Bleaching powder.

Methodology:

Iodometric method

Apparatus:

1. 250 ml cap. Conical flask

2. 250 ml cap.Volumetric flask

3. 10ml and 25 ml pipette

4. 50 ml Burette

5. Porcelain crucible with a glass rod

Reagents Used:

1.Standard Sodium thiosulphate solution of 0.025 N.

2. Potassium Iodide (KI) crystals.

3. Glacial Acetic Acid.

4. Starch indicator Solution.

5. Bleaching powder

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Percentage of chlorine available in a given sample of bleaching powder	_____

Discussion/Comments on Results:

Theory:

The chemicals (or) substances which are used for killing the pathogenic bacteria from water are known as disinfectants and the process is known as disinfection of water. Chlorine is heavier than air, greenish yellow colored and toxic gas. It is used in water and waste water treatment for disinfections to destroy pathogens and control nuisance causing micro-organisms and for oxidation. It is a strong oxidizing agent reacting with most of the elements and compounds. As an oxidant it is used in Iron and Manganese removal, destruction of taste and odour compounds and elimination of Ammonia, Nitrogen from water.

Bleaching powder is commonly used as disinfectant in small water treatment plants. Normally bleaching powder contains chlorine up to 35%. It is an unstable compound and hence it has to be stored carefully.

To find out the amount of available chlorine in bleaching powder, gets reduced with time, this test should always be conducted before adding bleaching powder to water.Iodometric method is considered as the standard one against other methods. It provides the means for standardizing the Chlorine water used in preparing temporary standard Chlorine, which will liberate free Iodine from Potassium iodide. The liberated Iodine is titrated with standard solution of Sodium thiosulphate using starch as indicator. The reaction is preferably carried out at pH 3-4.

Procedure:

1. Dissolve 250 mg of given bleaching powder in a 250 ml volumetric flask.

2. Take 25 ml of bleaching powder solution in a conical flask.

3. Add 5ml of Acetic acid (Glacial) add mix well. Note down the pH value (3-4).

4. Add 1 gram of KI (Potassium Iodide crystal) and mix well.

5. Titrate immediately with standard Sodium thisulphate solution till the light yellow color appears.
6. Add 1 ml of starch indicator the yellow color changes to dark blue.

7. Continue the titration till the blue color disappears. Note down the ml of $Na_2S_2O_3$ solution used (V).

Tabulation:

Flask No.	Sample taken in ml	Concentration of BP solution %	BP solution added	Chlorine added		(b) R-Cl in mg/l	Chlorine Demand (a-b) in mg/l
				200ml	1000ml (b)		
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							

Expt. No. 8   **Date:** _____

CHLORINE DEMAND

Aim of the Experiment:
Estimation of Chlorine demand in a given sample of water.

Methodology:
Iodometric method

Apparatus:

1. 500 ml cap. Conical flask
2. 250 ml cap. Volumetric flask
3. 10ml and 25 ml pipette
4. 50 ml Burette

Reagents Used:

1. Standard Sodium thiosulphate solution of 0.025 N and 0.01 N.
2. Potassium Iodide (KI) crystals.
3. Glacial Acetic Acid.
4. Starch indicator Solution.
5. Bleaching powdersolution.

Theory:

Chlorine is widely used for disinfection of water and wastewater to eliminate disease causing organisms, taste and odor. Since it is a powerful oxidizing agent and is cheaply available. The chlorine demand of water is the difference between the amount of chlorine applied and amount of free combined or total available chlorine at the end of contact period. The demand varies with amount of chlorine applied, pH and temperature. The smallest amount of residual chlorine considered significant is 0.1 mg/l.In small treatment plants bleaching powder is added as a disinfectant which obtain in the form of hydro chlorite of calcium which sterilize the water when chlorine is added to water it reacts with water as follows:

$Cl_2 + H_2O \longrightarrow$ HOCl (Hypochlorous acid) + HCl HOCl +
$OCl^- \longrightarrow$

The quantity of HOCl and OCl which is present in water is called available chlorine. The killing efficiency of HOCl is about 40 to 80 times more than that of OCl. It ruptures the cell membrane of microbes.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Chlorine demand of given sample of water	_____ mg/l

Discussion/Comments on Results:

Tabulation:

Flask No	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
FR										
IR										
FR-IR										
(b) R-Cl in mg/l										

Calculations:

1. Residual chlorine as CL in mg/l = $\dfrac{V \times N \times 35.45 \times 1000}{\text{ml of sample used}}$

2. Sample taken in ml = 200ml
   Concentration of BP solution (%) =
   BP solution added =
   Chlorine added

$$Ca(OCl)_2 \longrightarrow Ca + 2OCl \text{ (Hypochloride ions)}$$

$$OCl^- + H^+ \longrightarrow HOCl \text{ (Hypochlorous Acid)}$$
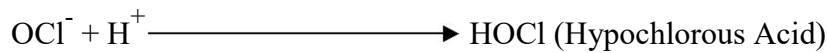
Hypochlorous Acid so formed kills the bacteria. The iodometric method considered as the standard against other method. The liberated iodine is titrated against the std. solution of Sodium Thiosulphate.

Procedure:

1. Prepare a bleaching powder solution of known concentration of Chlorine.
2. Measure 200ml of water sample for which Chlorine demand has to be found out in a series of 500 ml cap. conical flask of say 10 Nos.
3. Add 0.2 ml of bleaching powder solution to first flask and then 0.4 ml bleaching powder solution to second flack and so on in ascending order to the successive portion in series.
4. Mix the solution in each flask gently and allow for contact time of about 30 min. for potable water and suitably higher for polluted water and secondary effluents.
5. After the contact period add 5ml of Acetic acid(glacial) and 1 gm of KI crystals and mix.
6. Now add 1 ml of starch indicator to each flask. Blue color formation indicates the presence of excess Chlorine, no color indicates that Chlorine demand of water is not sufficient.
7. Titrate the sample with 0.01 N $Na_2S_2O_3$ solutiontill blue color disappears.
8. Record the ml of $Na_2S_2O_3$ solution consumed and note down the reading.

Observations:

1. Conical flask: 200ml of water sample with bleaching powder solution

2. Burette :   Standard sodium thiosulphate of 0.01 N

3. Indicators: Starch solution

4. End point: blue to colorless

Observations:

1. Conical flask: 201ml of sample from BOD bottle

2. Burette :   Standard sodium thiosulphate of 0.025 N

3. Indicators: Starch solution

4. End point: blue to colorless

Tabulation:

SL NO	BOTTLE NO.	VOL OF SAMPLE FOR TITRATION IN ml	BURRETTE READINGS			VOL. OF $Na_2S_2O_3$ USED
			FR	IR	FR-IR	
1.						
2.						
3.						

Calculations:

DO present in a given sample of water = $\underline{V \times 8 \times N \times 1000}$

ml of sample taken

= _____ mg/l

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	DO in a given sample	_____ mg/l

Discussion/Comments on Results:

Expt. No. 9   **Date:** _____

DISSOLVED OXYGEN

Aim of the Experiment:

To determine the quantity of Dissolved Oxygen present in a given sample of water.

Methodology:

Wrinkler's method (Azide modification)

Apparatus:

1. 300 ml capacity BOD bottle with stopper.
2. 500 ml capacity conical flask.
3. 100 ml measuring jar.
4. 50 ml Burette.
5. 10 ml pipette.

Reagents Used:

1. Manganese Sulphate solution.
2. Alkali Iodine solution (Azide).
3. Concentrated Sulphuric acid.
4. Standard Sodium Thiosulphate solution of 0.025N
5. Starch solution.

Theory:

All living organisms are dependent upon Oxygen in one or the other form to maintain their metabolic process the produces energy for growth and reproduction. The solubility if atmospheric oxygen in fresh water ranges from 14 mg/l @ 0 ^{o}C to about 7 mg/l @ 25 ^{o}C. Solubility varies directly with respect to the atmospheric temperature. It is one of the most important test that the environmental engineers use. Oxygen is a significant factor in the corrosion of Iron and stell, particularly in water distribution systems. The analysis of DO is a key test in water pollution control activities and waste treatment process control. The Winkler's test remains the most precise and reliable titrimeteric procedure for DO analysis. The test is based on the addition of divalent Manganese Sulphate solution followed by strong alkaline to water sample in a glass stopper bottle.

In the presence of Iodine ions and upon acidification, the oxidized Manganese prevents to the divalent state with the liberation of Iodine, equivalent to the original DO content in the sample. The iodine is then titrated with a standard solution of Sodium thiosulphate.

Procedure:

1. Collect the water sample in 300ml BOD bottle and take care to avoid contact of sample with air.
2. Add 1ml of Manganese Sulphate solution by a pipette. Insert the stopper, remove the excess liquid by spilling out by inverting the bottle and mix thoroughly.
3. Add 1ml of Alkali Iodine (Azide) Solution by a pipette. Insert the stopper, remove the excess liquid by spilling out by inverting the bottle and mix thoroughly. Wait for few minutes.
4. After the precipitate is settled, remove the stopper and immediately add 1 ml of concentrated H_2SO_4.
5. Re-stopper and mix by gentle inversion until the precipitate is dissolved.
6. Measure 201ml of the sample for BOD bottle into a clean conical flask.
7. Add 2-4 drops of starch indicator. The Solution color turns dark blue.
8. Titrate the sample with 0.025 N $Na_2S_2O_3$ till blue color disappears.
9. Note down the volume of Sodium Thiosulphate used (V).

Reactions:

$MnSO_4 + 2KOH \longrightarrow Mn(OH)_2 + K_2SO_4$

$Mn(OH)_2 + 2H_2SO_4 \longrightarrow \qquad + 3H_2O$

$Mn(SO_4)_2 + 2KI \longrightarrow MnSO_4 + K_2SO_4 + I_2$
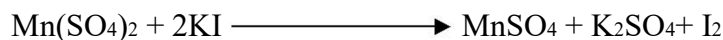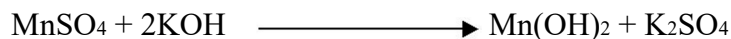
The quantity of Iodine liberated by these reaction is equivalent to the quantity of Oxygen present in the sample

Tabulation:

SL NO	SAMPLE USED	METER READING NTU
1.	Sample 1	
2.	Sample 2	

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Turbidity of given sample of water	_____ NTU

Discussion/Comments on Results:

Expt. No. 10   **Date:** _____

DETERMINATION OF TURBIDITY

Aim of the Experiment:

To determine the turbidity of given sample of water.

Methodology:

Turbidimetric method (photoelectric measurement)

Apparatus:

Digital Nephelo Turbidity Meter

Theory:

Turbidity is the measure of resistance of water to allow the light pass through it. It is caused by the pressure of suspended and colloidal matter such as clay,finally microscopic organisms,turbidity should be clearly understood to be expression of the optical property of sample which causes the light to be scattered and absorbed rather than transmitted in straight line through the sample the amount and character of the turbidity will depend on the type of soil over which the water has seen and velocity of water very finely divided clay settle very slowly may require month for complete settlement. The standard method of determination of turbidity has been based on the Jackson Candle Turbidity Meter. However the lower turbidity which can be measured directly on this instrument is 25 units with treated water generally falling in the range of 0-5 units. To estimate the turbidity of such sample can be made using digital nephelo turbidity meter.

Principle:

Turbidity measurement by the candle turbidity meter is based on the light path through suspension which just causes the image of the plane of standard candle to disappear that is to become indistinguishable against the general background illumination when the flame is viewed through the suspension. The longer light pass be the lower the turbidity. Measurements of turbidity using the photoelectric turbidity meter is based upon comparison of the intensity of light scattered by standard reference suspension under some conditions.

Results:

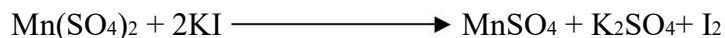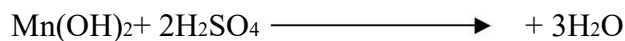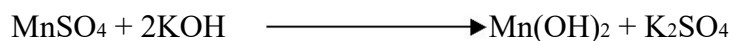| SL NO	PARAMETERS ANALYSED	RESULTS
1.	Turbidity of given sample of water	_____ NTU

Discussion/Comments on Results:

In the presence of Iodine ions and upon acidification, the oxidized Manganese prevents to the divalent state with the liberation of Iodine, equivalent to the original DO content in the sample. The iodine is then titrated with a standard solution of Sodium thiosulphate.

Procedure:

1. Collect the water sample in 300ml BOD bottle and take care to avoid contact of sample with air.

2. Add 1ml of Manganese Sulphate solution by a pipette. Insert the stopper, remove the excess liquid by spilling out by inverting the bottle and mix thoroughly.

3. Add 1ml of Alkali Iodine (Azide) Solution by a pipette. Insert the stopper, remove the excess liquid by spilling out by inverting the bottle and mix thoroughly. Wait for few minutes.

4. After the precipitate is settled, remove the stopper and immediately add 1 ml of concentrated H_2SO_4.

4. Re-stopper and mix by gentle inversion until the precipitate is dissolved.

5. Measure 201ml of the sample for BOD bottle into a clean conical flask.

6. Add 2-4 drops of starch indicator. The Solution color turns dark blue.

7. Titrate the sample with 0.025 N $Na_2S_2O_3$ till blue color disappears.

8. Note down the volume of Sodium Thiosulphate used (V).

Reactions:

$$MnSO_4 + 2KOH \longrightarrow Mn(OH)_2 + K_2SO_4$$

$$Mn(OH)_2 + 2H_2SO_4 \longrightarrow \quad + 3H_2O$$

$$Mn(SO_4)_2 + 2KI \longrightarrow MnSO_4 + K_2SO_4 + I_2$$

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	pH value of the given sample using pH meter	_____
2.	pH value of the given sample using pH paper	_____

Discussion/Comments on Results:

Expt. No. 11   **Date:** _____

<u>DETERMINATION OF pH</u>

Aim of the Experiment:

To determine the pH of given sample of water.

Methodology:

Using pH paper, pH indicator and pH meter.

Apparatus:

Digital pH Meter

Reagents Used:

Turbidity free distilled water

Theory:

pH is a negative logarithm of the reciprocal of the hydrogen ion concentration. The pH is a negative logarithm of the reciprocal of hydrogen ion concentration. The pH scale is used to express the degree of acidity or alkalinity with the middle value (pH) corresponds to the exact neutrality at 25^0C. The pH value represents the instantaneous hydrogen ion activity i.e., electrode system which is the most accurate method and free of interference. The pH paper is specially prepared one which will show the variation in pH with different color changes. Thus this method is suitable firmly rough estimation.

Procedure:

1. <u>Using pH meter.</u>
a) Follow the manufacture operating instruction.
b) Calibrate the instrument with buffer solution (known pH solutionof pH 4 to 7)
c) Dip the electrodes in the unknown water sample and note down the instrument reading which will give the direct pH value of the unknown sample.

2. <u>Using pH indicator (universal pH indicator of pH 4 to 11)</u>
a) Follow the instructions given in the indicator bottle.
b) Pipette out 10ml of given water sample into a small test tube.
c) To this add 0.2ml of universal pH indicator solution and mix well
d) Compare the color developed with the color chart and note down the pH values.
This method is also an approximate method of estimation of pHranging to 4 to 11.

3. <u>pH paper (pH0 to 10)</u>
a. Dip the pH paper in the given water sample.
b. Compare the color developed with that of water given in the wrapper of the pH paper booklet.
c. Note down the pH of sample along with it.

Calculations:

1. Total solids:

Empty weight of evaporation dish (W_1) = _____ grams Weight of evaporation dish after evaporating the sample (W_2) = _____ grams

Total solids (TS)

= $\underline{(W_2) - (W_1) \times 10^6}$ mg/1 ml
of sample

2. Dissolved solids:

Empty weight of evaporation dish (W_1) = _____ grams Weight of evaporation dish after evaporating the sample (W_2) = _____ grams

Dissolved solids (DS)

= $\underline{(W_2) - (W_1) \times 10^6}$ mg/1 ml
of sample

3. Suspended solids:

Suspended solids(SS) = Total solids – Dissolved solids = _____ grams

Expt. No. 12

Expt. No. 12   **Date:** _____

DETERMINATION OF SOLIDS

Aim of the Experiment:

To determine the amount of total solids, dissolved solids and suspended solids in a given sample of water.

Methodology:

Sample methods involves, evaporation, ignition of dried material present in the given water sample.

Apparatus:

1. Evaporating dishes made of porcelain, silicon.
2. Steam bath
3. Hot air oven
4. Desiccator
5. Weighing balance
6. Filter paper
7. Measuring jar
8. Funnel etc

Procedure:

Part A: Total solids

1. Clean the evaporating dish and place it in an oven at 103 ^{o}C for 15 mins.
2. Remove the evaporating dish and place it in desiccator.
3. Note down the initial weight of the evaporating dish (W_1 grams).
4. Take a known volume of sample i.e., 20 ml or 50 ml in the evaporating dish and evaporate the sample.
5. Cool the dish in a desiccator and note down the finial weight (W_2 grams).

Part B: Dissolved solids

1. Clean the evaporating dish and place it in an oven at 103 ^{o}C for 15 mins.
2. Remove the evaporating dish and place it in desiccator.
3. Note down the initial weight of the evaporating dish (W_1 grams).
4. Take a known volume of filtered sample i.e., 20 ml or 50 ml in the evaporating dish and evaporate the sample.
5. Cool the dish in a desiccator and note down the finial weight (W_2 grams).

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Total solids	_____ mg/1
2.	Dissolved solids	_____ mg/1
3.	Suspended solids	_____ mg/1

Discussion/Comments on Results:

Part C: Suspended solids

This test can be done for wastewater samples. Suspended solids can be calculated by subtracting the dissolved solids from total solids.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Electrical conductivity of the given sample	_____ ms @ 30 ºC

Discussion/Comments on Results:

Expt. No. 13   **Date:** _____

DETERMINATION OF ELECTRICAL CONDUCTIVITY

Aim of the Experiment:
To determine the electrical conductivity of a given sample of water.

Methodology:
Instrumental method

Apparatus:
Conductivity meter

Reagents used:
KCl, Distilled water

Procedure:
1. Connect the conductivity cell, temperature probe to the socket in the meter.
2. Connect the meter to the power supply and switch on the supply.
3. Allow the meter for few seconds.
4. Dip the conductivity cell and temperature probe in standard KCl solution.
5. Calibrate the instrument after placing the standard KCl solution.
6. Remove the conductivity cell and temperature probe from the solution and rinse it with distilled water. Wipe the conductivity cell and temperature probe with tissue paper.
7. Dip the conductivity cell and temperature probe in water sample and note down the reading in the display.

Tabulation:

SL NO	SAMPLE USED	INDICATOR USED	BURRETTE READINGS			VOL. OF FAS USED
			FR	IR	FR-IR	
1.	Waste water sam[ple	Ferrion red indicator				

Calculations:

$$COD = \frac{(V_1 - V_2) \times N \times 8 \times 1000}{ml \ of \ sample \ taken} \ \ mg/l$$

Expt. No. 14   **Date:** _____

CHEMICAL OXYGEN DEMAND TEST

Aim of the Experiment:

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

Methodology:

Titration and Open reflux method.

Apparatus:

1. COD digester
2. 250 ml conical flask
3. 50 ml burette
4. Pipettes, etc.

Reagents used:

1. Potassium dichromate solution
2. Ferrous ammonium sulphate (FAS) solution of 0.25 N.
3. Ferrion red indicator
4. Mercuric sulphate
5. Concentrated Sulphuric acid

Theory:

Chemical oxygen demand (COD) is the oxygen required for chemical oxidation of organics & inorganic impurities by strong oxidizing agents like potassium dichromate ($K_2Cr_2O_7$) under acidic conditions. The basis for the COD test is that nearly all organic compounds can be fully oxidized to carbon dioxide with a strong oxidizing agent under acidic conditions.

The major advantage of COD test is the short time required for determination of total O_2 required for oxidation. COD test requires 03 hours instead of 3 or 5 days as needed for measurement of BOD. The COD test is much more useful than BOD test for estimating strength of certain industrial effluents of both organic type (pesticide industries) and inorganic type (metallurgical industries) which contain toxic chemicals.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Chemical Oxygen Demand of the given sample	_____ ms @ 30 ºC

Discussion/Comments on Results:

Procedure:

1. Take 20 ml of sample in a clean conical flask.
2. Add 10 ml $K_2Cr_2O_7$ to conical flask and add 30 ml of concentrated H_2SO_4 slowly containing Ag_2SO_4 and mix thoroughly. Add 0.4 gram or pinch of mercuric sulfate ($HgSO_4$). Mix the contents thoroughly.
3. Transfer the sample into the glass digester of COD apparatus and connect the air vessel.
4. Reflux the sample for 2½ hours at a temperature of $\pm100^0C$. Allow it for some time for cooling to room temperature.
5. Dilute the sample with distilled water to make it up to 100 ml.
6. Titrate excess of $K_2Cr_2O_7$ with 0.25 N FAS as a titrant with 2 to 3 drops of Ferroin indicator until color changes from greenish blue to wine red indicating the end point of titration.
7. Note down the volume of titrant used for given sample as V1 ml and for distilled water (blank) as V2 ml.

Calculations:

1. Initial DO present in diluted wastewater (W_0) = _____ mg/l
2. Final DO present in diluted wastewater (W_3 = _____ mg/l
   3. Initial DO present in distilled water (D_0) = _____ mg/l
   4. Final DO present in distilled water (D_3) = _____ mg/l

$$\frac{(W_o - W_3) - (D_o - D_3) \text{ x Volume of BOD bottle 300 ml}}{\text{ml of sample taken in BOD bottle}}$$

BOD_3 of the sample = (Initial DO – Final DO) x Dilution ratio in mg/l

Expt. No. 15   **Date:** _____

BIOCHEMICAL OXYGEN DEMAND TEST

Aim of the Experiment:

To determine the Biochemical Oxygen Demand (BOD) of a given wastewater sample.

Apparatus:

1. BOD bottle (300 ml Capacity)
2. Incubator to control the temperature
3. Volumetric flasks
4. 50 ml Burette
5. 500 ml conical flask

Reagents:

1. Manganese Sulphate solution.
2. Alkali Iodine solution (Azide).
3. Concentrated Sulphuric acid.
4. Standard Sodium Thiosulphate solution of 0.025N
5. Starch solution.

Theory:

Microorganisms such as bacteria are responsible for decomposing organic matter. When organic matter such as dead plants, leaves, grass clippings, manure, sewage, food waste is present in a wastewater, the aerobic bacteria will start the oxidation of these wastes. When this happens, much of the available Dissolved Oxygen (DO) is consumed by aerobic bacteria, robbing other aquatic organisms of the oxygen they need to live. The biochemical oxygen demand is measure of oxygen utilized by aerobic micro-organisms during biological oxidation of organic matter. Generally, when BOD levels are high, there will be low DO levels.

Organic matter + O_2 $\longrightarrow$ CO_2 + new bacterias + H_2O + Heat Drinking water must have a BOD of less than 01 mg/l and the water is considered fairly up to 03 mg/l of BOD, but when the BOD value $\geq$ 05 mg/l the water is doubtful in purity.

Ordinary domestic sewage may have a BOD of 200 mg/l. As per CPCB standards the treated or untreated sewage to be discharged into surface water body must a have of BOD of less than 30 mg/l

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Biological Oxygen Demand of the given sample	_____ mg/1

Discussion/Comments on Results:

Procedure:

Part A: Dilution

1. Place the desired volume of distilled water in a 05 liter conical flask. Aeration is done by bubbling compressed air through distilled water.
2. Add 01 ml of manganous sulfate ($MgSO_4$) solution, 01 ml of calcium chloride ($CaCl_2$) and 01 ml of ferric chloride ($FeCl_3$) solution for every liter of distilled water.
3. In the case of the wastewater samples, which are not expected to have sufficient bacterial population, add seed to the distilled water. Generally 2 ml of settled sewage is sufficient for 1000 ml of distilled water as seed.
4. Highly acidic or alkaline samples are to be neutralized to pH of around 7.0.
   5. Add 2 or 3 ml of sodium thiosulfate ($Na_2S_2O_3$) to destroy residual chlorine if any.
   6. Take sample as under:

   Strong wastes: 0.1, 0.5, or 1%
   Settled domestic sewage: 1.0, 2.5, or 5%
   Treated effluents: 5.0, 12.5 or 25%
   River water: 25 to 100%

7. Dilute the sample with distilled water and mix the contents well.

Part B:Titration

1. Take samples in 02 BOD bottles of 300 ml capacity.
2. Fill another 02 BOD bottles with distilled water (blank).
3. Immediately find initial DO of 01 bottle with distilled water in it and 01 bottle with diluted wastewater sample in it by modified Azide method or Winkler's method (same procedure as used in DO determination).

Incubate the remaining 02 bottles by keeping them in an incubator for 5 days (120 hours) at 20^0C or for 3 days (72 hours) at 27^oC and find out the final DO of the distilled water and water/wastewater samples by modified Azide method or Winkler's method.

Tabulation:

SL NO	SAMPLE	ALUM DOSAGE (800 ml)	ALUM DOSAGE (1000 ml)	INITIAL TURBIDITY	RESIDUAL TURBIDITY	TURBIDITY REMOVED
1.	Sample given in the lab					
2.						
3.						
4.						
5.						
6.						

Nature of Graph:

Expt. No. 16   **Date:** _____

JAR TEST

Aim of the Experiment:
To determine the optimum coagulant dosage for a given water sample.

Methodology:
Conducting jar test with variable dosage of alum solution.

Apparatus:

1. Jar test apparatus
2. Turbidity meter
3. 5ml, 10 ml pipette
4. 1000 ml beakers
5. Turbidity meter

Theory:

The available raw water must be treated and purified before supplied to domestic, industrial, commercial or any use. The extent of treatment required to be given to water depends upon the characteristics and quality of water. Water must be made safe and good in appearance. Turbidity of surface water is due to the presence of suspended matter and other impurities that will not settle in simple sedimentation. It is necessary to add coagulant to the water to remove the turbidity. The best of treatment can be first decided in lab on a small scale and can be implemented in treatment plants. Most common apparatus used for this purpose is Jar test apparatus.

Principle:

Coagulants are used in water treatment plant to:

☐ Remove natural, suspended and colloidal matter.
☐ Remove material, which don't settle by plain sedimentation.
☐ To assist in filtration.

With careful control of coagulant dosage, very clean water can be obtained after the coagulation. Jar test is a simple device which with helps in determining the required optimum coagulant dosage. The jar test device consist of jars (5 to 6) provided with stirrers and peddles. Sample will be taken in jar or beakers varying dose of coagulant will be added simultaneously to all the jars.

Results:

SL NO	PARAMETERS ANALYSED	RESULTS
1.	Optimum coagulant dosage	_____ mg/1

Discussion/Comments on Results:

Procedure:

1. Measure the initial turbidity of the given sample of water.
2. Take 6 beakers of 1000ml and measure the sample of water in all 6 beakers and place them in the jar test apparatus. (Check the position of the stirrers etc. switch on the instrument and adjust the speed of the peddles to 100-200 rpm).
3. Add 1 ml of standard alum solution to the 1st beaker, 2ml to the 2nd and add the alum solution in increasing order to the next beakers.
4. Rapid mix the sample for 1 minute.
5. Bring down the speed to 30-40 rpm and allow for 9 minutes.
6. Switch off the instrument after 9 minutes and allow the liquid for 20 minutes for settling.
7. Take 50 ml of supernatant from each jar without disturbing the sample.
8. Measure the turbidity of all the samples with the help of turbidity meter.
9. Repeat the steps from 1-8 with higher dosage of alum if necessary.

Expt. No. 17   **Date:** _____

MOST PROBABLE NUMBER (MPN) DETERMINATION

Aim of the Experiment:
To determine the Total Coliform (MPN) in given sample.

Methodology:
Bacteriological Analysis - Multiple Tube Fermentation Technique.

Apparatus:
Autoclave, BOD incubator, Durham's tubes, Borosil glasswares etc.

Reagents:
Mackonkey broth solution

Theory:
The Most Probable Number (MPN) technique is an important technique in estimating microbial populations in soils, waters, and agricultural products. The methodology for the MPN technique is dilution and incubation of replicated cultures across several serial dilution steps. MPN is the number, which indicates the bacteria density, which is most likely to be present in water. The MPN is based on the application of laws of statics to the results of test and is, therefore more accurate than Coliform Index (CI) method. The MPN index of coliform micro-organism for potable water shall be less than 1 in 100 ml of water sample as per BIS: 10500-1991.

There are many advantages of using the MPN technique. MPN methodology results in more uniform recovery of a microbial population. Furthermore, the detection of organisms through process-related attributes often results in the recovery of mixed populations with similar functional roles in soils. For a more detailed study, the mixed populations can be separated into individual colonies. Another advantage of MPN techniques is that, unlike direct quantitative procedures, it measures only live and active organisms. Microscopic techniques sometimes confuse live and dead cells. Despite the numerous advantages of using MPN methodology there are a few disadvantages to the method. MPN procedures tend to require more labor and materials than microscopic procedures. Also, MPN estimates often have a lower order of precision than do well-replicated direct counts. There are two techniques to find most probable number of confirmed microorganisms in a water sample. They are (a) multiple tube fermentation technique and (b) membrane filter technique. Multiple tube fermentation technique is more useful than membrane filter technique because of its applicability to almost all kinds of waters.

Results:

Total Coliform MPN/100ml =   (No. of +ve tubes x 100)

$$\overline{(m \sqrt{\ } f \text{ sample in -ve tubes}) \times (ml \text{ of sample in all tubes})}$$

Discussion/Comments on Results:

Procedure:

1) Wash thoroughly and sterilize all the glasswares before use.
2) Prepare the Broth solution of both double strength (DS) & single strength (SS) as per the specifications
3) Arrange the series of 10.0, 1.0 & 0.1 ml combination. (05 test tubes for each combination).
4) Place 10 ml broth (DS) solution in each of first 05 test tubes, 10 ml (SS) solution in remaining 10 test tubes.
5) Drop Durham's tube (inverted) in each test tube and plug the test tube with cotton.
6) Again sterilize the solution and other glasswares.
7) Add 10 ml representative sample in each of the first 05 (DS) tubes, 1.0 ml sample in second 5 (SS) tubes and 0.1 ml sample in remaining 5 (SS) tubes and plug the test tube with cotton.
8) Incubate the tubes for initial 24 hours for preliminary observation of +ve tubes and another 24 hours for confirmative observation at about $35 \square 2^0 C$.
9) Note down the +ve test tubes from each combination and refer the MPN table for the final value.
10) If the combination is not matching with the table, use the following formula,