



ATRIA INSTITUTE OF TECHNOLOGY

Affiliated to VTU

LABORATORY MANUAL

**18CVL67-Environmental Engineering
Laboratory**

2021-2022

DEPARTMENT OF CIVIL ENGINEERING

AKSB Campus, 1st Main Road, Anand Nagar,

Hebbal, Bengaluru 560024

SYLLABUS

Course Title: Environmental Engineering Laboratory
As per Choice Based Credit System (CBCS) scheme SEMESTER:VI

Subject Code	18CVL67	IA Marks	40
Teaching Hours/Week (L:T:P)	(0:2:2)	Exam Marks	60
Total Number of Lecture Hours	40	Exam Hours	03
CREDITS –02		Total Marks- 100	

Course objectives: This course will enable students,

1. To learn different methods of water & waste water quality
2. To conduct experiments to determine the concentrations of water and waste water
3. To determine the degree and type of treatment
4. To understand the environmental significance and application in environmental engineering practice

Experiments	Teaching Hours	Revised Bloom's Taxonomy (RBT) Level
1. Preparation chemical solutions required for analysis and sampling methodologies	02 Class	L1,L2,L3
2. Determination of pH, Conductivity, TDS and Turbidity	02 Class	L1,L2,L3
3. Determination of Acidity and Alkalinity .	02 Class	L1,L2,L3
4. Determination of Calcium, Magnesium and Total Hardness		
5. Determination of Dissolved Oxygen	01 Class	L1,L2,L3
6. Determination of BOD.	01 Class	L1,L2,L3
7. Determination of Chlorides	01 Class	L1,L2,L3
8. Determination of percentage of % of available chlorine in bleaching powder sample, Determination of Residual Chlorine and chlorine demand	02 Class	L1,L2,L3
9. Determination of Solids in Sewage: i) Total Solids, ii) Suspended Solids, iii) Dissolved Solids, iv) Volatile Solids, Fixed Solids v) Settleable Solids	02 Class	L1,L2,L3
10. Determination of optimum coagulant dosage using Jar test apparatus	01 Class	L1,L2,L3
11. Determination Nitrates and Iron by spectrophotometer	02 Class	L1,L2,L3
12. Determination of COD(Demonstration)	Demonstration	L1,L2,L3
13. Air Quality Monitoring (Demonstration)	Demonstration	L1,L2,L3
14. Determination of Sound by Sound level meter at different locations (Demonstration)	Demonstration	L1,L2,L3
		L1,L2,L3
		L1,L2,L3

Course Outcomes: After studying this course, students will be able to:

1. Acquire capability to conduct experiments and estimate the concentration of different parameters.
2. Compare the result with standards and discuss based on the purpose of analysis.
3. Determine type of treatment, degree of treatment for water and wastewater.
4. Identify the parameter to be analyzed for the student project work in environmental stream.

Program Objectives:

1. Evaluation of the test results and assesses the impact on water and waste watertreatment.
2. Train student to undertake student project work in 8th semester in the field of environmentalengineering.

Question paper pattern:

Two experiments shall be asked from the above set

One experiment to be conducted and for the other student should write detailed procedure

Reference Books:

1. Lab Manual, ISO 14001 Environmental Management, Regulatory Standards for Drinking Water and Sewage disposal
2. Clair Sawyer and Perry McCarty and Gene Parkin, "Chemistry for Environmental Engineering and Science", McGraw-Hill Series in Civil and Environmental Engineering

LIST OF EXPERIMENTS

S. No.	Experiment
1	To measure the pH value of water sample using digital pH meter
2	Determination of acidity
3	Determination of alkalinity
4	Determination of total hardness of water sample
5	Determination of calcium and magnesium hardness
6	Dissolved oxygen test
7	Biochemical oxygen demand
8	Determination of chloride
9	Determination of percentage of available chlorine in bleaching powder
10	Determination of residual chlorine
11	Determination of solids in sewage
12	Turbidity determination by nephelometer
13	Determination of optimum dosage of alum using jar test apparatus
14	Determination of sodium and potassium by flame photometer
15	Determination of nitrates by spectroscopic method
16	Determination of iron by phenanthroline method
17	Chemical oxygen demand
18	Air quality monitoring (ambient, stack monitoring , indoor air pollution)
19	Determination of sound by sound level meter at different location

LAB SAFETY INSTRUCTIONS

- Always prohibit eating, drinking, or smoking near samples, sampling locations, and in the laboratory.
- Keep sparks, flames, and excessive heat sources away from samples and sampling locations.
- Label adequately any sample known or suspected to be hazardous because of flammability, corrosiveness, toxicity, oxidizing chemicals, or radioactivity, so that appropriate precautions can be taken during sample handling, storage, and disposal.
- Students **without shoes will not be allowed in the lab.**
- Bring lab coat and a permanent marker to lab.
- Always wear full sleeves clothes.
- Wear safety glasses and gloves when recommended.
- Leave bags and coats in designated areas. Bring only the essentials to the lab bench.
- No eating, drinking, playing, or applying cosmetics (including hand lotion, etc.).
- Never use broken or chipped glassware. Place broken glassware in specially marked containers.
- Mouth pipetting is forbidden.
- Hands should be washed after contact with hazardous materials and before leaving the lab.

EXPEREMENT NO: 01
TO MEASURE THE pH VALUE OF WATER SAMPLE USING
DIGITAL pH METER

AIM: To measure the pH of the given sample

APPARATUS REQUIRED/ CHEMICALS REQUIRED:

- 1) Digital pH meter and beakers (250ml)
- 2) Distilled water
- 3) Buffer solutions pH-4, pH-7 and pH-9.2
- 4) Unknown solution

PREPARATION OF SOLUTIONS:

Buffer solution, for pH 4 one tablet of pH 4 + 100 ml distilled water pH 7

one tablet of pH 7 + 100 ml distilled water pH 9.2

one tablet of pH 9.2 + 100 ml distilled water.

THEORY:

Every phase of water supply and waste water treatment, e.g. acid base, neutralization, water softening, precipitation, coagulation, disaffection and corrosion control is pH dependent. More – over many chemical and biochemical reactions are depending upon pH. So pH measurement is an important aspect

pH of a solution is defined as the negative logarithm (to the base 10) of hydrogen ion concentration. It may be mathematically stated as

$$\text{pH} = -\log_{10} [\text{H}^+]$$

Similarly, pOH of a solution is defined as $\text{pOH} = -\log_{10} [\text{OH}^-]$ For

any dilute solution $\text{pH} + \text{pOH} = 14$

The pH value of any solutions ranges from 0-14. The pH scale is give as follows:

pH value: 0 1 2 3 4 5 6 7 8 9 10 11 12 13 14

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Acidic Basic

PRINCIPLE:

The basic principle of the experiment is determination of the activity of the hydrogen ions by potentiometric measurement using a glass electrode and a reference electrode. The glass electrode method is widely accepted because, the glass electrode is much easier to use and is less likely to be poisoned in highly impure water. Glass electrode consists of HCl or a buffer solution in contact with an internal reference electrode. Upon immersion of this in a solution, the outer bulb surface become hydrated and exchanges Na^+ ions for H^+ ions build up a surface layer of H^+ ions. This, together with the repulsion of anions by fixed negatively charged silicates produces at the glass solution interface a potential which is a function of H^+ ion activity in solution.

PROCEDURE:

A) Instrument calibration:

Connect the three pin plug to 230 V mains. Remove the electrode from storage solution and rinse with distilled water. Dry the electrode gently, blotting with a soft tissue paper. Take the buffer solution in a clean glass beaker. Dip the electrode in the solution and set the temperature of the solution using “temperature C knob”. Adjust ‘standardize’ knob so that the display reads the exact the value of the buffer solution. Standardize the instrument with electrode immersed in a buffer solution, within 2 pH units of sample ph. Remove the electrode from a buffer solution, rinse thoroughly with distilled water and finally dry it. Immerse the electrode in another buffer solution, having the pH 2 units higher than that of the experimental solution. Now the reading should be within 0.1 units for the pH of the second buffer. Otherwise, look for trouble with the electrode; switch off the instrument when not in use.

B) Sample analysis:

Now, immerse the electrode in a solution of unknown pH, taken in a beaker. Establish the equilibrium between the electrode and sample. Stirring the sample to ensure homogeneity (1 min). Switch on the instrument and read the pH. Again immerse in a fresh portion of the same sample and read the pH. In this pH meter, pH scale may be read off either in pH numbers or in mili volts for which a separate arrangement has been kept.

RESULT:

pH of the given sample of water = Sample No. 1 =

Sample No.2 =

CONCLUSION:

EXPEREMENT NO: 02

DETERMINATION OF ACIDITY

AIM: To determine acidity of the given sample.

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 pH

The CO₂ and bicarbonates (carbonic acid) present and contribute CO₂ acidity in the sample can be neutralized completely by continuing the titration to pH 8.2.

APPARATUS:

1. Burette
2. Conical flask
3. Pipettes.

REAGENTS:

1. Standard sodium hydroxide (0.02N)
2. Phenolphthalein indicator.
3. Methyl orange indicator.
4. Sodium thiosulphate (0.1N)
5. Carbon dioxide free distilled water.

PROCEDURE:

1. Take 100 ml of the given sample in a conical flask.
2. Add 1 drop of 0.1N sodium thiosulphate solution to remove the residual chlorine if present.
3. Add 2 drops of Methyl orange, the sample turns Red.
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 100ml 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).

CALCULATIONS:

$$\text{Mineral acidity due to mineral acids (as CaCO}_3\text{) (mg/l) = } \frac{V1 \times 10}{\text{Volume of Sample taken}}$$

$$\text{Total acidity (as CaCO}_3\text{) (mg/l) = } \frac{V2 \times 100}{\text{Volume of Sample taken}}$$

OBSERVATIONS:

Sample details	Volume of the sample (ml)	Methyl orange indicator			Phenolphthalein indicator		
		Initial	Final	NaOH used (ml)	Initial	Final	NaOH used (ml)

RESULTS:

Mineral acidity (mg/l) =

Total acidity as (CaCO₃) = Mineral acidity + CO₂ acidity.

CONCLUSION:**ENVIRONMENTAL SIGNIFICANCE:**

EXPEREMENT NO: 03

DETERMINATION OF ALKALINITY

AIM: To determine alkalinity of a given sample

APPARATUS:

- 1) Burette
- 2) Conical flask
- 3) Pipette

REAGENTS:

- 1) Standard Sulphuric acid (0.02N).
- 2) Phenolphthalein indicator.
- 3) Methyl orange.
- 4) Sodium thiosulphate (0.1N).

THEORY:

Alkalinity of water is defined as measure of its capacity to neutralize the acids.

Alkalinity of water is due to,

- 1) Hydroxides, carbonates, bi-carbonates of elements and ammonia.
- 2) Salts of weak acids and strong base.
 - a) Barites, silicates and phosphates.
 - b) Salts of organic acids and formic acids or acids.
 - c) Salts of acetic proteomics hydro-sulphuric acid
 - d) Algae utilize the free and combine CO₂ present in natural water during photosynthesis highly alkaline water is unsuitable for domestic industrial and agricultural purpose. Hence determination of alkalinity is important and not is measured volumetrically.

PRINCIPLE:

Alkalinity is determined by titrating against 0.02N sulphuric acid using phenol phallic having pH >8.3 titration made in 2 steps.

In 1st step titration is carried out until ph reaches 8.3, at that point there will be de- coloration of phenolphthalein indicator which shows complete neutralization of hydroxyl anion (OH⁻) and half of carbonate ion (CO₃⁻).

In 2nd step, titration is carried out by addition of methyl orange, ph further reaches to 4.5. At this point there is sharp change from yellow to orange of methyl orange indicator this indicates the total alkalinity.

INTERFERENCE FACTOR:

Free available residual chlorine markedly affects the Indicator colour response. The addition of minimal volumes of sodium thiosulphate eliminates this Interference. Substances such as salt of weak organic or Inorganic acids present in large amount may interfere. Oils and greases may also interfere by coating the electrode. Colored or turbid samples may interfere in end point. Analyze such samples by potentiometric titration.

PROCEDURE:

- 1) Take 100ml of given sample in conical flask.
- 2) Add 1 drop of 0.1N sodium thiosulphate solution to remove the free residual chlorine if present.
- 3) Add 2 drops of phenolphthalein indicator. The sample turns to pink.
- 4) Titrate it against 0.02N standard sulphuric acid till the solution turns colourless.
- 5) Note down the volume of sulphuric acid added (V₁).
- 6) Add 2 drops of methyl orange indicator the sample turns yellow.
- 7) Resume the titration till the colour of the solution turns pink.
- 8) Note down the volume of sulphuric acid added (V₂).

Value of P and T	Alkalinity due to		
	OH ⁻	CO ₃ ⁻	HCO ₃ ⁻
P=0	0	0	T
P<1/2 T	0	2P	T-2P
P=1/2 T	0	2P	0
P>1/2 T	2P-T	2T-2P	0
P=T	T	0	0

OBSERVATION:

Trial no	Sample details	Vol of sample taken	Phenolphthalein indicator			Methyl orange indicator		
			Initial	Final	H ₂ SO ₄ Used(ml)	Initial	Final	H ₂ SO ₄ Used(ml)
01	Tap water	100ml						
02								
03								
04	Sample	100ml						
05								
06								

CALCULATION:

1) Phenolphthalein alkalinity (P) mg/lit as CaCO₃

$$P = V_1 \times \text{Normality of H}_2\text{SO}_4 \times 50000 / \text{Vol of sample taken}$$

2) Total alkalinity (T) mg/lit as CaCO₃

$$T = (V_2 + V_1) \times \text{Normality of H}_2\text{SO}_4 \times 50000 / \text{Vol of sample taken}$$

For tap water

1) Phenolphthalein alkalinity (P) = _____ mg/l as CaCO₃

2) Total alkalinity (T) = _____ mg/l as CaCO₃.

For sample

1) Phenolphthalein alkalinity (P) = _____ mg/l as CaCO₃

2) Total alkalinity (T) = _____ mg/l as CaCO₃

Sample	Value of P and T	Alkalinity due to		
		OH ⁻	CO ₃ ⁻	HCO ₃ ⁻
Tap water	P=0	0	0	T=
Given sample	P>1/2T	2P-T	2T-2P	0

RESULT:

TAP WATER: 1) Phenolphthalein alkalinity (P) = _____ mg/l as CaCO₃

2) Total alkalinity (T) = _____ mg/l as CaCO₃

BORE WATER: 1) Phenolphthalein alkalinity (P) = _____ mg/l as CaCO₃

2) Total alkalinity (T) = _____ mg/l as CaCO₃

CONCLUSION:

EXPERIMENT NO: 04

DETERMINATION OF TOTAL HARDNESS OF WATER SAMPLE

AIM: To determine the total hardness of given water sample

APPARATUS:

Burette, conical flask, pipette

REAGENTS USED:

- 1) Ammonia buffer solution
- 2) Erichrome black T indicator
- 3) Standard EDTA solution as titrate (0.1M)

THEORY:

Hardness in water is that characteristics which prevents the formation of sufficient lather or foam, when such hardness are mixed with soap. Hardness is a measure of the ability of water to cause precipitation of insoluble calcium and magnesium salts of higher fatty acids from soap solutions.

Hardness is defined as the characteristics of water which represents the total concentration of calcium and magnesium ions expressed as CaCO_3 and hence hardness is always reported as molar equivalent of CaCO_3 in mg/lit. Hardness of water is not a specific element but variable accounted by a complex mixture of cat ions and anions.

Relative abundance of cations and anions causing hardness.

Cations causing hardness	Anions causing anions
Ca^{+2}	HCO_3^{-}
Mg^{+2}	SO_4^{2-}
Sr^{+2}	Cl^{-}
Fe^{+2}	NO_3^{-}
Mn^{+2}	CO_3^{2-}

Hardness is as satisfactory as soft water from human consumption point of view. Due to adverse action with soap the use for cleaning purpose hard water use is generally avoided or used after treatment. It leads to scale formation in heaters and beakers, causes corrosion,

incrustation of pipes.

Hardness scale: water are commonly classified in terms of the degree of hardness as follows

Degree of hardness	mg/l as CaCO ₃
Soft	0-75
Moderately hard water	50-100
Hard water	150-300
Very hard water	>300

TYPES OF HARDNESS:-

Hardness is classified with respect to,

- 1) Metallic cations
- 2) Anions associated with metallic ions.
 - 1) With respect to metallic ions
 - a) Calcium hardness
 - b) Magnesium hardness
 - 2) With respect to the anions associated with metallic ions
 - a) Carbonate hardness
 - b) Non- Carbonate hardness

When water with temporary hardness is boiled CO₂ gas escapes out and the unstable calcium carbonate gets precipitated magnesium carbonate, being fairly soluble in water don't get removed by boiling the temporary hard water, therefore do cause deposition of calcium scales in sackers.

If sulphates, chlorides and nitrates of calcium or magnesium are present in water they cannot be removed at all by simple boiling and therefore such water require special treatment for softening. Such hardness is known as permanent hardness or non-carbonate hardness.

Carbonate hardness and non-carbonate hardness can be calculated by using the following relationship

- a) When alkalinity < total hardness
- b) Carbonate hardness = alkalinity

Non carbonate hardness = total hardness – carbonate hardness

c) When alkalinity > total hardness

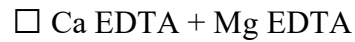
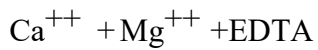
Carbonate hardness = total hardness

Non carbonate hardness is absent.

Note: the above relationship holds good only when alkalinity and hardness are both expressed in terms of CaCO_3 .

PRINCIPLE:

Under alkaline conditions EDTA forms soluble complexes with calcium and magnesium ions at pH i.e. $\text{pH} = 10 \pm 0.1$. A small amount of erichrome black T indicator is added to the water containing calcium and magnesium ions at pH of 10 ± 0.1 water becomes wine red in colour. If EDTA is used as titrate against the water sample containing calcium and magnesium ions it will form complexes calcium and magnesium and solution turns to blue colour which is the end point.



PROCEDURE:

1) TOTAL HARDNESS

1. Take 100ml of sample in a clean conical flask.
2. Add 1ml of ammonia buffer solution.
3. Add 1 pinch of Erichrome black-T indicator colour of the solution turns to wine red.
4. Titrate against std EDTA solution till the colour changes to wine red to clear blue note down the burette reading (A-B).
5. Total hardness in mg/lit as $\text{CaCO}_3 = ((A-B) \times 1000) / (\text{ml of sample taken})$.

2) PERMANENT HARDNESS

1. Boil the sample continuously until all the CO_2 gets expelled from the surface.
2. Cool the sample.
3. Take 100ml of sample in a clean conical flask.
4. Add 1ml of ammonia buffer solution.
5. Add 1 pinch of Erichrome black-T indicator colour of the solution turns wine red.
6. Titrate against std EDTA solution till the colour changes to wine red to clear blue

note down the burette reading (A-B).

7. Permanent hardness in mg/lit as CaCO₃ = ((A-B)×1000)/ (ml of sample taken).

OBSERVATION AND CALCULATION:

1) TOTAL HARDNESS:

Trial No.	Volume of Sample	Sample Details	Burette reading		
			Initial reading	Final reading	ml of std EDTA used
01					
02					
03					

$$\text{Total hardness in mg/l as CaCO}_3 = \frac{(\square - \square) \times 1000}{\text{NS of caNpse uced}}$$

$$= \text{as CaCO}_3.$$

2) PERMANENT HARDNESS:

Trial no	Volume of sample taken (ml)	Sample details	Observation		
			Initial reading	Final reading	EDTA used
01					
02					
03					

$$\text{Permanent hardness in mg/lit as CaCO}_3 = \frac{(\square - \square) \times 1000}{\text{NS of caNpse uced}}$$

$$= \text{mg/l as CaCO}_3$$

$$\text{Temporary hardness} = \text{total hardness} - \text{permanent hardness} = \text{mg/l as CaCO}_3$$

RESULT:

- 1) Total hardness of given sample = _ mg/l as CaCO₃
- 2) Permanent hardness of given sample = _ mg/l as CaCO₃
- 3) Temporary hardness of given sample = _ mg/l as CaCO₃

CONCLUSION:

EXPERIMENT 5

DETERMINATION OF CALCIUM AND MAGNESIUM HARDNESS

AIM: To determine the calcium and magnesium hardness of given water sample.

APPARATUS:

Burette, Conical flasks, Pipettes

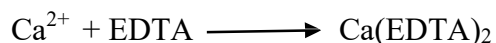
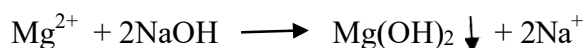
REAGENTS:

1N NaOH, Murexide, EDTA (0.1M)

PRINCIPLE:

Under highly alkaline condition (pH=12-13), Magnesium precipitates as magnesium hydroxide and calcium forms complexes with EDTA in presence of indicator which combines with calcium only.

REACTIONS:



PROCEDURE:

CALCIUM HARDNESS:

1. Take 100ml of water sample in a clean conical flask.
2. Add 1ml of 1N NaOH solution into the sample.
3. Add 1 pinch of murexide into the solution.
4. Titrate it against Std. EDTA till the colour changes from pink to purple. Note down the burette reading (A-B)

OBSERVATION AND CALCULATION:

Trial no	Volume of sample taken (ml)	Sample details	Observation		
			Initial reading	Final reading	EDTA used
01					
02					
03					

$$\text{Calcium hardness in mg/l as CaCO}_3 = \frac{(\square - \square) \times 10}{\text{NS of CaNpSe used}}$$
$$= \underline{\hspace{2cm}} \text{mg/L as CaCO}_3$$

Magnesium hardness in mg/l as CaCO₃ = Total hardness – Calcium hardness

RESULT: Calcium hardness of given sample =

Magnesium hardness of given sample =

CONCLUSION:

EXPERIMENT NO: 06

DISSOLVED OXYGEN TEST BY WINKLER'S METHOD OR MODIFIED AZIDE METHOD

AIM: To find the quantity of dissolved oxygen present in the given sample.

APPARATUS:

BOD bottle (capacity 300ml), sampling device for collection of sample, burette, pipette and measuring jar.

REAGENTS:

- 1) Manganese sulphate
- 2) Alkali iodide Azide reagent
- 3) Starch indicator
- 4) Concentrated sulphuric acid
- 5) Standard sodium Thiosulphate (0.025N)

PRINCIPLE:

Oxygen present in sample oxidizes the divalent manganese to its higher valency which precipitates as a brown hydrated oxide after addition of NaOH and K .Upon acidification manganese reacts to divalent static acid and liberates iodine from K equivalent to BOD content in the sample. The liberated iodine is titrated against $\text{Na}_2\text{S}_2\text{O}_3$ (0.025N) using starch as indicator. If oxygen is absent in sample the MnSO_4 reacts with alkali to form white precipitate $\text{Mn}(\text{OH})_2$.

PROCEDURE:

- 1) Take a clean and dry BOD bottle and collect 300ml of water sample in it.
- 2) Tap the neck sample of the BOD bottle to expel air bubble if any.
- 3) Add 2ml of manganese sulphate and 2ml of alkali iodide azide solution to the BOD bottle. The tap water of the pipette should be below the liquid level while adding the above said solution.
- 4) Re-stopper with care to exclude air bubble.
- 5) Mix the content properly by repeatedly inverting the bubble 10-15 times.
- 6) If oxygen is present then the manganese ion gets converted into a brown colour

manganese oxide (MnO₃). After taking and allowing sufficient limit for all oxygen to react, the chemical ppt is allowed to settle thereby having a clear liquid at the top portion.

- 7) A 2ml of conc. sulphuric acid and mix the sample completely by re-stopping the bottle and inverting it.
- 8) A dark yellow colour solution is obtained which is immediately titrated against sodium this sulphate solution by taking 203ml in a conical flask until the colour changes to pale yellow (strew yellow).
- 9) Now add starch as an indicator, the colour of solution turns to blue, continue titration till the colour disappears.

TABULATION AND CALCULATION:

Trial no	Volume of sample	Burette reading		Sodium thiosulphate rundown
		Initial reading	Final reading	
01	200			
02	200			
03	200			

$$\text{Dissolved oxygen (mg/l)} = \frac{V_{\text{of Sodium thiosulphate}} \times \text{Normality of sodium thiosulphate} \times 8000}{V_{\text{of Sample}}}$$

Dissolved oxygen (mg/l) for tap water= mg/l.
 Dissolved oxygen (mg/lit) for given water sample= mg/l.

RESULT:

Dissolved oxygen present in the given tap water sample= mg/lit.
 Dissolved oxygen present in the given water sample= mg/lit.

CONCLUSION:

EXPERIMENT NO: 07

BIOCHEMICAL OXYGEN DEMAND

AIM: To determine the biochemical oxygen demand in the given sample of water.

APPARATUS:

- 1) BOD bottle 300 capacity.
- 2) Incubator, to be controlled at $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
- 3) Burette,
- 4) Pipette and
- 5) Measuring jar.

REAGENTS:

- 1) Phosphate buffer.
- 2) Magnesium sulfate.
- 3) Calcium chloride.
- 4) Ferric chloride.
- 5) Sodium Thiosulfate solution 0.025N.
- 6) Additionally all reagents listed in DO estimation.

THEORY:

BOD is defined as the amount of oxygen required by microorganisms while stabilizing biologically decomposable organic matter in a waste under aerobic conditions. The BOD test is widely used to determine

- 1) The pollution load of waste water,
- 2) The degree of pollution in lakes and streams at any time and their self-purification capacity and
- 3) Efficiency of waste water treatment methods.

Since the test is mainly a bio-assay procedure, involving measurement of oxygen consumed by bacteria while stabilizing organic matter under aerobic conditions, it is necessary to provide standard conditions of nutrient supply, pH, absence of microbial growth inhibiting substances and temperature. Because of the low solubility of O_2 in water, strong wastes are always diluted to ensure that the demand does not increase the available O_2 . A mixed group of organisms should be present in the sample: if not, the sample has to be seeded artificially.

Temperature is controlled at 20°C. The test is controlled for 5 days as 70 to 80% of the waste is oxidized during this period.

PROCEDURE:

PREPARATION OF DILUTION WATER:

- 1) Aerate the required volume of distilled water in a container by bubbling compressed air for about 15 min to attain DO saturation. Try to maintain the temperature near 20°C.
- 2) Add 1ml of phosphate buffer, magnesium sulphate, calcium chloride and ferric chloride solution for each liter of dilution water. Mix well.
- 3) In the case of the wastes which are not expected to have sufficient bacterial pollution, add seed to the dilution water. Generally, 2ml settled sewage is considered sufficient for 100ml of dilution water.

DILUTION OF SAMPLE

- 1) Neutralize the sample to pH around 7.0 if it is highly alkaline or acidic.
- 2) Take 50ml of the sample and acidify with addition of 10ml 1+1 acetic acid. Add about 1kg KI. Titrate with sodium thiosulphate 0.025N using starch indicator. Calculate the volume of sodium thiosulphate required per ml of the sample and add accordingly to the sample to be tested for BOD.
- 3) Samples having high DO content i.e. DO 9mg/l, due to either algal growth or some other reason, reduce the DO content by aerating the samples.
- 4) Make several dilutions of the pretreated sample so as to obtain about 50% depletion of DO in dilution water but not less than 2mg and the residual O₂ after 5 days of incubation should not be less than 1mg/lit. prepare dilutions as follows
- 5) Siphon out seeded dilution water in a measuring cylinder or volumetric flask half the required volume. Add the required quantity of carefully mixed sample. Dilute to the desired volume by siphoning dilution water and mix well.
- 6) The following dilutions are suggested:

0.1% to 1%	Strong trade waste
1% to 5%	Raw or settled sewage
5% to 25%	Treated effluent
25% to 100%	River water

Keep 1 bottle for determination for the initial DO and incubate 2 bottles at 20°C for 5 days.

See that the bottles have a water seal. Prepare a blank in duplicate by siphoning plain dilution water (without seed) to measure the O₂ consumption in dilution water. Fix DO of the bottles kept for immediate DO determination and blank by adding 1ml MnSO₄ followed by 1ml alkali iodide azide solution as described in the estimation of DO. Determine DO in the sample and in the blank on initial day and after 5 days.

OBSERVATION AND CALCULATIONS:

Initial DO

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
10% dilution			
20% dilution			

DO (10%) = _____ ml

DO (AV %) = _____ ml

Five-Day DO

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
10% dilution			
20% dilution			

C₅= _____ ml

D₅ (10%) = _____ ml

D₅ (AV %) = _____ ml

CALCULATION:

BOD mg/l= ((D₅-D₀)-(C₅-C₀))/ (ml of sample used in decimals)

Where: D₅ and D₀ = DO in sample after 5 days and initial day respectively

C₅ and C₀=DO in blank after 5 days and initial day respectively

RESULT:

BOD of the sample = _____ mg/l.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT NO: 08

DETERMINATION OF CHLORIDE BY ARGENTOMETRIC METHOD OR MOHR'S SALT METHOD

AIM: To determine the chloride in the given water sample.

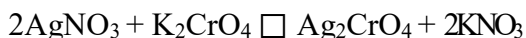
APPARATUS:

Burette, conical flask and measuring jar

PRINCIPLE:

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

REACTIONS:



THEORY:

Chloride associated with sodium exerts salty taste, when its concentration is more than 250mg/l. Chloride do not cause any human health hazards but corrodes concrete the extracting calcium in the form of calcite MgCl_2 in water i.e. magnesium chloride generates hydro chloride acid after which is also highly corrosive and create problems in suckers.

REAGENTS:

- 1) Potassium chromate indicator solution.
- 2) Standard silver nitrate solution (0.0282 N).

PROCEDURE:

- 1) Take 100ml of the sample in the conical flask.
- 2) Add 2 to 3 drops of potassium chromate indicator into the solution and shake well.
The solution turns to pale yellow.
- 3) Titrate it against standard silver nitrate solution (0.0141N).
- 4) Continue the titration till the end point of pale yellow to brick red is reached.
- 5) Note down the reading (i.e. volume of silver nitrate added-A)

6) Continue the same procedure for 100ml distilled water and note down the volume of silver nitrate-(B).

FORMULA:

$$\text{Cl (mg/l)} = \frac{((A-B) \times (\text{normality of AgNO}_3 \times 35.45 \times 1000))}{(\text{ml of sample})}$$

OBSERVATION AND TABULATION:

End point: reaction completion point colour changes from yellow to brick red.

Sample details	Trial no	Vol of sample taken (ml)	Observation		AgNO ₃ solution used
			Initial reading	Final reading	
	1				
	2				
	3				

CALCULATION:

$$\text{Cl (mg/l)} = \frac{\text{[Blank space for calculation]}}{\text{[Blank space for calculation]}}$$

Sample 01=

Sample 02=

RESULT:

Chloride content in given water

Sample 01: _____ mg/l.

Sample 02: _____ mg/l.

CONCLUSION:

EXPERIMENT NO: 09
DETERMINATION OF PERCENTAGE OF AVAILABLE CHLORINE
IN BLEACHING POWDER

AIM: To determine the quantity of available chlorine in a given bleaching powder sample.

APPARATUS:

Conical flask, Burette, Pipette and Volumetric flask.

REAGENTS:

Bleaching powder, Glacial acetic acid, Potassium iodide crystals or powder, Standard sodium thiosulphate (0.1N), Starch indicator solution.

THEORY:

Bleaching powder is nothing but chlorinated lime or CaOCl_2 (calcium oxychloride). This compound is a white amorphous powder with a pungent smell of. When freshly made, it contains about 30-35% of available chlorine. It is however an unstable compound and on exposure to air, light and moisture it rapidly loses its chlorine content. Bleaching powder is used for treating small water surplus swimming pools and it can also be used as emergency disinfectant.

FORMULA:

Available chlorine in bleaching powder = $\frac{(A-B) \times 0.1 \times 35.45 \times 1000}{\text{volume of sample}}$

PROCEDURE:

- 1) Measure exactly 5gm of given bleaching powder and dissolve it completely in 1000 ml of distilled water.
- 2) Take 100ml of solution and add 1g of KI crystals and about 5ml of glacial acetic acid. Leave the sample for 10 min for reaction.
- 3) Titrate the solution against standard sodium thiosulphate of 0.1N until the colour turns to pale yellow.
- 4) Add 2 to 3 drops of starch indicator solution and continue the titration till the solution turns blue to colourless. Note down the burette reading (A).
- 5) Repeat the titration for distilled water (B).

OBSERVATION AND CALCULATION:

SI No	Sample	Burette reading		ml of sodium thiosulphate used	Available Chlorine
		Initial reading	Final reading		
01	Bleaching				
02	powder solution				
03	Distilled water				

Available chlorine in bleaching powder = $((A-B) \times 0.1 \times 35.45 \times 1000) / (\text{volume of sample})$
= mg/l.

% of chlorine = $((\text{available chlorine}) / 1000) \times 100$
= %

RESULT:

Available chlorine in bleaching powder sample = mg/l.

% of chlorine sample = %

CONCLUSION:**ENVIRONMENTAL SIGNIFICANCE:**

EXPEREMENT NO: 10

DETERMINATION OF RESIDUAL CHLORINE

AIM: To determine the residual chlorine available in given water sample.

APPARATUS:

Beaker, pipette, conical flask and volumetric flask.

REAGENTS:

KI, Acetic acid, Sodium thiosulphate (0.0025N) and Starch indicator.

THEORY:

DOSAGE OF CHLORINE: The amount of chlorine required for the water depends upon the amount of inorganic impurities and organic impurities present in it when chlorine is added to water it first reacts with inorganic impurities like Sr^- , Mn^{2+} , NO^{2-} , Fe^{2+} , etc which converts the chlorine into chloride. After this point excess chlorine is consumed by ammonia to form chloramines. After this point chlorine will react with organic impurities present in water. The chlorine used in all the above reaction represents chlorine demand of water once after chlorine demand is satisfied the chlorine will appear to be free chlorine (residual chlorine). The function of free residual chlorine is to immediately kill the pathogens where as Cl will provide long term germicide effect.

PROCEDURE:

1. Take 100ml of sample in a conical flask and add a pinch of potassium iodide.
2. Add 5ml of acetic acid and allow the reaction to complete.
3. Titrate the sample against 0.0025N of sodium thiosulphate solution until the yellow colour disappears
4. Add 1ml of starch solution, blue colour appears then continue the titration until the blue colour disappears (A-B).

FORMULA:

Residual chlorine = $((A-B) \times 0.0025 \times 35.45 \times 1000) / (\text{volume of sample})$.

OBSERVATION AND CALCULATION:

SL NO	Sample taken	Burette reading		Difference	Residual chlorine in (mg/l)
		Initial reading	Final reading		
01	Sample 01				
02	Sample 02				

Residual chlorine = $((A-B) \times 0.0025 \times 35.45 \times 1000) / (\text{volume of sample})$.

= mg/l.

RESULT:

Residual chlorine in the given sample

= mg/l.

CONCLUSION:**ENVIRONMENTAL SIGNIFICANCE:**

EXPERIMENT: 11
DETERMINATION OF SOLIDS IN SEWAGE
TOTAL SOLIDS

AIM: To determine the total solids of a given sample.

APPARATUS:

Evaporating dish, oven and desiccators.

PRINCIPLE:

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

PROCEDURE:

- 1) Take 100ml of well mixed sample and pour it into evaporating dishes which is already been heated in an oven at 103°C for removing the moisture and desiccated for balancing the temperature and weighed (W_1).
- 2) Heat the sample until it is dried (24hrs).
- 3) Take out the evaporating dish ported in a desiccators and take out the final reading (W_2).

OBSERVATION:

Weight of the empty dish, W_1 = g.

Weight of the sample with dish (oven dried), W_2 = g.

Volume of the sample taken, V = g.

CALCULATION:

Initial weight of the evaporating dish (W_1) = g.

Final weight of the evaporating dish (W_2) = g.

Total solids = $((W_2 - W_1) \times 1000) / (\text{volume of sample})$. =mg/l.

RESULT:

Total solids of a given sample = mg/l.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

TOTAL SUSPENDED SOLIDS

AIM: To find the total suspended solids in a given sample.

APPARATUS:

Evaporating dish, wattman filter paper and Hot air oven.

PRINCIPLE:

A well-mixed sample is filtered through a filter paper and the residue retained on the filter is dried to a constant weight 103°C. The increase in weight of filter paper represents the total suspended solids.

PROCEDURE:

- 1) Take a wattman filter paper.
- 2) Place in an oven and heat it at 103⁰C to remove the moisture. Take a filter paper from the oven placed in desiccators to balance the temperature and take the initial weight (W₁).
- 3) Pour known volume of well mixed sample to the filter paper.
- 4) Once after the completion of filtration take the filter paper place it in an the oven and heat it for 103⁰ C for 1 hour.
- 5) Take out the filter paper from oven and place it in the desiccators to balance the temperature and note down the final reading (W₂).

OBSERVATION AND CALCULATION:

Empty weight of filter paper, W₁= _____ g.

Weight of filter paper + suspended solids, W₂= _____ g.

Volume of sample= _____ ml.

Total suspended solids = ((W₂-W₁)*1000*1000)/ (volume of sample).

= _____ mg/l.

RESULT:

Total suspended solids = _____ mg/l.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

TOTAL DISSOLVED SOLIDS

AIM: To find the total dissolved solids in a given sample.

APPARATUS:

Crucible, filter paper and hot air oven.

PROCEDURE:

Take an evaporating dish, heat it in the oven to remove the moisture, place it in a desiccator to balance the temperature and take the initial weight W_1 . Take known volume of well mixed sample and filter it from a filter paper which is previously dried and weighed evaporating dish. The filtrate left over in an evaporating dish is dried at 103°C and desiccated for balancing the temperature and weight take the final weight W_2 .

OBSERVATION AND CALCULATIONS:

Empty weight of evaporating dish, $W_1 =$ _____ g.

Weight of sample with dish after filtration (after oven drying), $W_2 =$ _____ g

Volume of sample = _____ ml

Total dissolved solids = $((W_2 - W_1) \times 1000 \times 1000) / (\text{volume of sample})$.

= _____ mg/l.

RESULT:

Total dissolved solids = _____ mg/l.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

TOTAL FIXED AND VOLATILE SOLIDS

AIM: To find out Total fixed and volatile solids of the given sample

PRINCIPLE:

Total volatile solids and fixed solids are determined as residue remaining after evaporation, drying at 103⁰ C and ignition at 600⁰C.

APPARATUS:

1. Evaporating dish.
2. Oven 103⁰C
3. Muffle furnace 600⁰C
4. Desiccators
6. Water Bath

PROCEDURE:

1. A clean porcelain dish is ignited in a muffle furnace and after partial cooling in air, it is cooled in a desiccators and weighed (W₁).
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 100⁰C on water bath, followed by drying in oven at 103⁰C for 1 hour.
3. Dry to a constant weight at 103⁰C, cool in desiccator and weighed (W₂).
4. Ignite the residue on evaporation at 600⁰C in the muffle furnace to constant weight in 10 to 15 min.
5. Allow the dish to cool and moisten the ash with a few drops of distilled water.
6. Dry to constant weight at 104⁰C, cool in a desiccators and weighed (W₃).

CALCULATIONS:

Total solids (mg/l) = ((W₂-W₁)×1000×1000)/ (volume of sample).

$$= \text{mg/l.}$$

Total volatile solids (mg/l) = Total solids-Fixed solids.

$$= \text{mg/l.}$$

Total fixed solids (mg/l) = $((W_3 - W_2) \times 1000 \times 1000) / (\text{volume of sample})$.

= mg/l.

OBSERVATIONS:

Type of solids	Sample details	Volume of sample, ml	Weight of empty dish (mg)	Weight of empty dish+ Residue (mg)	Residue (mg/l)

RESULTS:

The amount of Total, fixed and volatile solids of the given sample is= mg/l

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

TOTAL SETTLEABLE SOLIDS

AIM: To find out Total settleable solids of the given sample.

PRINCIPLE:

The particles in suspensions whose specific gravity greater than that of water will settle under quiescent conditions.

APPARATUS:

1. Imhoff cone.
2. Holding device.

PROCEDURE:

1. Gently fill the Imhoff cone with the thoroughly well mixed sample usually one liter and allow it to settle.
2. After 45 minutes, gently rotate the cone between hands to ensure that all solids adhering to the sides are loosened.
3. Allow the solids to settle for 15 minutes more, to make up for a total period of 1 hour.
4. Read the volume of the sludge which has settled in the apex.
5. Express the results in ml settleable solids per liter of sample per hour.

CALCULATIONS:

Total settleable solids (mg/l) = (ml of solids x 1000)/ml of sample.

PRECAUTIONS:

1. The imhoff cones must be cleaned with a strong soap and hot water using a brush.
2. Wetting the cone with water before use, helps in preventing adherence of the solids to the sides.
3. The method is subjected to considerable in accuracy if the solids contain large fragments.
4. The determination of total settleable solids should be carried out soon after sampling in order to avoid errors through flocculation.

OBSERVATIONS:

Sample details	Volume of sample taken (ml)	Total settleable solids ml/l/hour

RESULTS: Total settleable solids of the given sample is = mg/l.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 12

TURBIDITY DETERMINATION BY NEPHLOMETER

AIM To determine the turbidity of the given sample.

APPARATUS:

Nephelo-turbidity meter, Beaker, Pipette

PRINCIPLE:

When light is passed through a sample having suspended particles, some of the light is scattered by particles. This scattering of light is generally proportional to the turbidity. The turbidity sample is of thus measured from the amount of light scattered by the sample, taking a reference with standard turbidity suspension.

THEORY:

Inhibition of light transmission due to the presence of suspended matter in water is termed as turbidity. It is a measure of optical property of light and depends on the presence of suspended matter like freely divided organic and inorganic matter silt, clay and microorganisms. The standard method used for turbidity measurement, is a nephelometric method and the result is expressed in NTU (Nephelometric Turbidity Unit).

PROCEDURE:

- 1) Switch on the instrument and allow it to warm up.
- 2) Take the standard suspension of the sample and calibrate the instrument.
- 3) After the calibration place the cell with the sample and note down the turbidity directly from the instrument.

TABLATIONS:

Sl No	Sample	Turbidity (NTU)
01	Distilled water	
02	Water sample	

RESULT:

The turbidity of the given sample is NTU.

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 13

DETERMINATION OF OPTIMUM DOSAGE OF ALUM USING JAR TEST APPARATUS

AIM: To determine the optimum dosage of coagulant required to treat the turbid waters.

PRINCIPLE:

Metal salts hydrolyze in presence of the natural alkalinity to form metal hydroxides. The divalent cations can reduce the zeta- potential, while the metal hydroxides are good absorbents and hence remove the suspended particles by enmeshing them.

APPARATUS:

1. Jar test apparatus.
2. Beakers.
3. Turbidity meter
4. pH meter

REAGENTS:

Alum solution- Dissolve 1 g of fresh alum in 1000 ml of distilled water.

PROCEDURE:

1. Measure the turbidity of given sample.
2. Take 1 litre of sample into each of 6 beakers.
3. Switch on the motor and adjust the speed of paddles to 100 rpm.
4. Add varying doses of alum solution i.e., 1 ml, 2ml, 3ml, 4ml, 5ml, 6ml to different beakers simultaneously.(The doses vary with turbidity in water sample).
5. Allow flash mix for 1 minute.
6. Reduce the speed of paddles to 40 rpm and continue mixing for 10 minutes.
7. Switch off the motor and allow 20 minutes for settling of flocs.
8. Collect the supernatant without disturbing the sediment and find the turbidity of each beaker.
9. Repeat the experiment with high doses of alum if satisfactory results are not obtained.
10. Plot a graph of turbidity removed v/s dosage of alum added.
11. Note the ideal (optimum) dose of the coagulant for excellent floc formation.

OBSERVATION:

Raw water turbidity (NTU) =

Raw water pH =

Raw water Alkalinity (mg/l) =

TABULATION:

Sl. No	Vol. of sample	Beaker No.	Weight of alum added	Initial turbidity NTU	Final turbidity NTU	turbidity removed
1						
2						
3						
4						
5						
6						

RESULT:

Ideal dosage of coagulant (mg/l) =

CONCLUSION:**ENVIRONMENTAL SIGNIFICANCE:**

EXPERIMENT: 14

DETERMINATION OF SODIUM BY FLAME PHOTOMETER

AIM: To determine the amount of Sodium present in the given sample solution.

PRINCIPLE:

Flame emission spectroscopy is a type of atomic emission spectroscopy. It is mostly applicable for analysis of alkali and alkali earth metals. In this spectroscopy, the sample solution of sodium salt is nebulized in to flame, which may produce solid residue upon solvent evaporation. This solid residue undergoes atomization and gives neutral atoms which may acquire thermal energy from flame and undergoes electronic excitation. Due to unstable nature of excited state, excited atoms come back to ground state by emission of absorbed energy as visible radiation. By measuring the wavelength and intensity of emitted radiation, we can do qualitative and quantitative analysis respectively.

CHEMICALS REQUIRED

1. Sodium chloride
2. Distilled water

APPARATUS REQUIRED:

1. Flame photometer
2. Volumetric flasks
3. Pipette

PROCEDURE:

Preparation of standard solutions for calibration curve:

Dissolve exactly 1.88 gm of Sodium chloride in water and make up to 1 liter. This contains 1mg per ml (1000 ppm).

Estimation of Sodium by flame photometer:

- 1) First, switch on the digital flame photometer followed by the air compressor with the required value (10 bar).
- 2) Open the gas from the gas cylinder (after the instrument is warmed up for 10 minutes). Initially allow the ion-free water (distilled water) to aspirate in to the flame and set the digital value as 100. Now the instrument is said to be calibrated. After this calibration of the instrument, no adjustment should be made. Introduce the solutions containing

different concentrations of Sodium chloride (2, 4, 6, 8, 10 μ g) to the flame and find out the intensity of emitted light of each solution.

- 3) Plot a calibration graph between concentration and intensity of NaCl solution which passes through the origin. Finally, introduce the sample of unknown solution containing sodium into the flame and find out the intensity of emitted radiation. From the intensity, the concentration of unknown solution can be determined.

DETERMINATION OF SODIUM:

Sl. No.	Concentration of NaCl Solution(ppm)	Flame intensity
1		
2		
3		
4		
5		
6		

The concentration of unknown sample =

RESULT:

The amount of Sodium in the given sampleppm

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

DETERMINATION OF POTASSIUM BY FLAME PHOTOMETRY

AIM: To determine the amount of potassium present in the given sample solution.

PRINCIPLE:

Flame emission spectroscopy is a type of atomic emission spectroscopy. It is mostly applicable for analysis of alkali and alkali earth metals. In this spectroscopy, the sample solution of potassium salt is neutralized in to flame, which may produce solid residue upon solvent evaporation. This solid residue undergoes atomization and gives neutral atoms which may acquire thermal energy from flame and undergoes electronic excitation. Due to unstable nature of excited state, excited atoms come back to ground state by emission of absorbed energy as visible radiation. By measuring the wavelength and intensity of emitted radiation, we can do qualitative and quantitative analysis respectively.

CHEMICALS REQUIRED:

1. Potassium chloride
2. Distilled water

APPARATUS REQUIRED:

1. Flame photometer
2. Volumetric flasks
3. Pipette

PROCEDURE:

Preparation of standard solutions for calibration curve:

Dissolve exactly 1.090gm of potassium chloride in water and make up to 1 liter. This contains 1mg per ml (1000 ppm).

Estimation of potassium by flame photometer:

- 1) First, switch on the digital flame photometer followed by the air compressor with the required value (10 bar).
- 2) Open the gas from the gas cylinder (after the instrument is warmed up for 10 minutes). Initially allow the ion-free water (distilled water) to aspirate in to the flame and set the digital value as 100. Now the instrument is said to be calibrated. After this calibration of the instrument, no adjustment should be made. Introduce the solutions containing different concentrations of potassium chloride (2, 4, 6, 8, 10 μ g) to the flame and find out the intensity of emitted light of each solution.

- 3) Plot a calibration graph between concentration and intensity of KCl solution which passes through the origin. Finally, introduce the sample of unknown solution containing sodium into the flame and find out the intensity of emitted radiation. From the intensity, the concentration of unknown solution can be determined.

DETERMINATION OF POTASSIUM:

Sl. No.	Concentration of KCl Solution(ppm)	Flame intensity
1		
2		
3		
4		
5		
6		

RESULT:

The amount of potassium in the given sample..... ppm

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 15

DETERMINATION OF NITRATES BY SPECTROSCOPIC METHOD

AIM: To find out nitrate nitrogen present in a given sample.

PRINCIPLE:

Nitrate reacts with phenol disulphonic acid and produces a nitro derivative which in alkaline medium develops a yellow colour. The colour produced follows the Beer's law and directly proportional to the concentration of nitrate present in the sample.

APPARATUS:

1. Spectrophotometer having a range of 300-700 nm.
2. Nessler tubes, capacity 100 ml.

REAGENTS:

Standard silver sulphate. , Phenol disulphonic acid (PDA) , Ammonium hydroxide, Stock nitrate solution, Standard nitrate solution.

PROCEDURE:

1. Take 50 ml of filtered sample in a Erlenmeyer flask.
2. Add a equivalent amount of silver sulphate to remove chlorides.
3. Heat slightly and filter the precipitate of AgCl.
4. Evaporate the filtrate in a porcelain disc to dryness.
5. Cool and dissolve the residue in 2 ml phenoldisulphonic acid and dilute to 50 ml.
6. Add 10 ml of liquid ammonia to develop the yellow colour.
7. Read the colour developed at 410 nm with a light path of 1cm.
8. Calculate the concentration of nitrate nitrogen from the standard curve.
9. Prepare the standard curve using suitable aliquots of standard nitrate solution in the range of 5 to 500mg NO₃-N/l following the procedures.

OBSERVATIONS:

RESULT: Nitrate nitrogen (mg/l) =

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 16

DETERMINATION OF IRON BY PHENANTHROLINE METHOD

AIM: To determine quantity of iron present in the given sample.

PRINCIPLE:

In the phenanthroline method, the ferric form of iron is reduced to ferrous form by boiling with hydrochloric acid and hydroxyl amine hydrochloride. The reduced iron chelates with 1, 10 phenanthroline at pH 3.2 to 3.3 to form a complex of orange-red colour. The intensity of colour is proportional to the concentration of iron and obeys Beer's law.

APPARATUS:

1. Nessler's reagent.
2. Conical flasks
3. Pipettes.

REAGENTS:

1. Hydrochloric acid
2. Hydroxylamine hydrochloride solution
3. Ammonium acetate buffer solution
4. Phenanthroline solution
5. Stock iron solution.
6. Standard iron solution (1ml=1 μg Fe)

PROCEDURE:

1. Pipette 10, 20, 30, and 50 ml of standard iron solution into 100ml conical flasks.
2. Add 1ml of hydroxylamine hydrochloride solution and 1ml of sodium acetate to each flask.
3. Dilute each to about 75 ml with distilled water.
4. Add 2ml of Phenanthroline solution to each flask.
5. Make up the contents of the each flask exactly to 100ml by adding distilled water and allow at least 10-15 minutes for maximum colour development.
6. For visual comparison, pour the solution in 100 ml Nessler's tubes and keep them in a stand.
7. Mix the sample thoroughly and measure 50 ml in to conical flask.
8. Add 2ml of con. HCL and 1ml of hydroxylamine solution and a few glass beads.

9. Boil the contents to half of the volume for dissolution of all the iron.
10. Cool the flask to room temperature and transfer the solution to 100 ml Nessler tube.
11. Add 10 ml of ammonium buffer solution and 2ml phenanthroline solution and dilute to 100 ml mark with distilled water. If the sample contains interference of heavy metals, add 10ml of phenanthroline instead of 2ml.
12. Mix thoroughly and allow at least 10 to 15 minutes for maximum colour development.
13. For visual comparison, match the colour of sample with that of the standards prepared in steps 1 to 6 above.
14. The matching colour standard will give the concentration of iron in the sample.

RESULT:

Iron content of the sample (mg/l) =

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 17 (Demonstration)

CHEMICAL OXYGEN DEMAND

AIM: To determine the chemical oxygen demand of the given sample.

APPARATUS:

- 1) Reflux apparatus consisting of a flat bottom 250 to 500ml capacity flask with ground glass joint and a condenser.
- 2) Burner or hot plate with temperature regulator.

REAGENTS:

- 1) Standard potassium dichromate 0.25N.
- 2) Sulphuric acid reagent.
- 3) Standard Ferrous Ammonium Sulphate 0.25N.
- 4) Ferroin indicator
- 5) Mercuric sulphate.

THEORY:

COD test determine the O₂ required for chemical oxidation of organic matter with the help of strong chemical oxidant. The test can be employed for the same purpose as the BOD tests taking into account its limitations.

The intrinsic limitation of the test lies in its inability to differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 5 hrs as compared to 5 days required for BOD test. Further, the test is relatively easy, gives reproducible results and is not affected by interferences as the BOD test.

PROCEDURE:

- 1) Place 0.3g mercuric sulphate in a reflux flask.
- 2) Add 20ml sample or an aliquot of sample diluted to 20ml with distilled water. Mix well.
- 3) Add pumice stone or glass beads followed by 10ml of std. Potassium dichromate.
- 4) Add slowly 30ml of Sulphuric acid containing Ag₂SO₄ mixing thoroughly. This slow addition along with swirling prevents fatty acid to

escape out due to high temperature.

- 5) Mix well, if the colour turns green, either take fresh sample with lesser aliquot or add more dichromate and acid.
- 6) Connect the flask to condenser. Mix the contents before heating, improper mixing will result in bumping and sample may be blown out.
- 7) Reflux for a minimum 20 min. Cool and then wash down the condenser with distilled water.
- 8) Keep it for cool, make the volume to 150ml by adding distilled water and then titrate against ferrous ammonium sulphate using Ferroin indicator. Sharp colours change from blue green to wine red indicates end point or completion of the titration.
- 9) Reflux blank in the same manner using distilled water instead of sample.

OBSERVATION AND CALCULATION:

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
Sample 01			
Sample 02			

$$\text{COD mg/l} = ((A-B) \times N \times 8000) / (\text{ml of sample}).$$

Where; A = ml Ferrous Ammonium Sulphate for blank.
B = ml Ferrous Ammonium Sulphate for sample.
N = normality of Ferrous Ammonium Sulphate.

RESULT: COD of sample= _____ mg/l

CONCLUSION:

ENVIRONMENTAL SIGNIFICANCE:

EXPERIMENT: 18 (Demonstration)

MONITORING OF RESPIRABLE PARTICULATE MATTER

AIM Sampling of respirable particulate matter in the ambient and indoor environment air and the demonstration of its concentration in various sizes.

APPARATUS

Environmental dust monitor with weather house (automatic sampler) for PM₁₀, PM_{2.5} and PM_{1.0}.

WORKING PRINCIPLE

The dust particles are measured by the physical principle of orthogonal light scattering. Here particles are illuminated by a laser light in an angle of 90 degree. All units of environmental dust monitor use light-scattering technology for single-particle counts. The scattered signal from the particle passing through the laser beam is collected at approximately 90° by a mirror and transferred to a recipient-diode. The signal of the diode is feed, after a corresponding reinforcement, to a multi-channel size classifier. This pulse height analyzer then classifies the signal high of each channel. These counts of each channel are converted each minute in a mass distribution from which the different PM values derive.

1. Sampling location guidelines

Sampling location should be located depending upon the objective of measurement campaign and be kept at an altitude depending upon the type of study region (roadways, industrial area, disposal sites, residential area, etc). Generally it is kept at a height of about 3m to 10m from the ground level and sufficiently away from the disturbance of direct obstacles from the source under consideration.

2. Sampling frequency guidelines

Sampling is carried out for various purposes. The regular monitoring campaign of national ambient air quality includes measurement of particulate matter typically for 24 hours at least twice a week making about 104 samples a year.

3. Steps for sampling

- a. Prepare a sampling assembly.
- b. Set the time constant depending upon the required averaging period.
- c. Instrument can be switch on and it will display concentration values in $\mu\text{g}/\text{m}^3$.
- d. Simultaneously instrument will start recording the concentration values in the memory card.

MONITORING OF GASES AND PARTICULATES IN AMBIENT AIR

AIM To measure the ambient concentrations of gases and particulate matter by using High Volume Sampler (HVS).

APPARATUS

- a. High volume sampler (HVS)
- b. Whatman filter paper
- c. Impingers

PRINCIPLE

PM₁₀ and TSPM are measured by passing air at flow rate of about 1 lpm through high efficiency cyclone which retains the dust particles greater than 10 micron size and allow only fines (less than 10 micron particles) to reach the glass microfibre filter where these particles are retained. The instrument provides instantaneous flow rate and the period of operation (on- time) for calculation of air volume passed through the filter. Amount of particulates collected is determined by measuring the change in weight of the cyclone cup and filter paper.

REAGENTS FOR GASEOUS POLLUTANTS

- N Sodium tetra-chloratemercurate (SO₂)
- Sodium hydroxide and sodium arsenite (NO₂)

PROCEDURE

i. For particulates

- a. Perform leak check of the instrument before starting the sample.
- b. Filter paper need to be inspected for pin holes.
- c. Filter conditioning need to be done at 20-25°C temperature and less than 50% Relative Humidity.
- d. Never fold filter completely.
- e. Do not touch filters by dirty hands always use disposable hand gloves.
- f. Under take regular cleaning of key components of the machine.
- g. Ensure stable power supply to the machine. Do not leave loose contact of supply wire to the machine.
- h. Always fill up distilled water in manometer assembly.
- i. Do not switch on and off machine using Timer Switch.
- j. Clean impinge and rotameter regularly and also clean manifold once in two months.
- k. Do not take flow reading immediately after switching on the machine. Give 5 minute for flow stabilization and for heat up the blower components.
- l. Always attach a new weighed cyclone cup with every filter change.
- m. Do not switch on machine without filter paper.

- n. If machine is not expected to be operated within 48 hrs drain out the manometer water and store machine with water in the manometer tank.
- o. Do not run machine during rain in open atmosphere.

ii. For gaseous

The increasing general awareness of atmospheric pollution and its hazards to the health and well-being of industrial workers, educational buildings, offices etc., is bound to result in greater stress on accurate, reliable and frequent assessment of work place pollution and worker-exposure. Use additionally impinge tray with HVS sampler simultaneously sample gaseous pollutants.

INDOOR AIR QUALITY MONITORS

AIM To understand how to operate the instrument and also know the basic knowledge of indoor air quality (IAQ) monitor.

APPARATUS

Indoor air quality monitor (automatic sampler) for carbon monoxide (CO), carbon dioxide (CO₂), temperature and humidity.

INDOOR AIR QUALITY MONITOR

With 90% of our time spent indoors, determining the quality of the air we breathe indoors is essential for good health and productivity. The IAQ monitor key indoor air quality indicators including CO₂, humidity, temperature and CO. Should these measurements fall outside recognized guidelines; further tests can be made to suggest an appropriate course of action. For example, ventilation studies show that as room temperatures rise above 75°F(24°C) the ability of occupants to concentrate can drop by up to 50% and high levels of carbon dioxide will indicate poor ventilation that results in drowsiness and perceived stuffiness. Both situations are very common and seriously affect productivity. Over-ventilation wastes energy and results in increased building running costs. The Surveyor range has been designed with the user in mind. Minimal training is required to use the instruments as the intuitive menu system and display provide step-by-step guides for each operation that are updated when smart probes are plugged in.

STEPS FOR SAMPLING

- a. Prepare a sampling assembly.
- b. Set the time constant depending upon the required averaging period.
- c. Instrument can be switch on and it will display concentration.
- d. Simultaneously instrument will start recording the concentration values in the memory card.
- e. Using data transfer cable (ie. RS232 cable) can download data from instrument to personal computers.

EXPERIMENT: 19 (Demonstration)

DETERMINATION OF SOUND BY SOUND LEVEL METER AT DIFFERENT LOCATION

AIM Determination of Sound by Sound level meter at different location

THEORY

Noise level measurement procedure are processes which are followed while monitoring sound level or acoustic energy level in specified area. These days annoying noise levels have started to become a major threat to public health. Noise in simple terms can be defined as unwanted or unpleasant sound which disturbs the environment and has a significant impact on the quality of life. We all very well know how exposure to high noise level can result in hearing impairment, headache, sleep disturbance and impaired task performance.

NOISE MEASUREMENT

Three techniques are used to measure noise in the work place and community environment

- (1) Personal sound exposure meter or also known as dosimeter
- (2) Sound Level Meter
- (3) Tape recorders, data recorders and level recorders

TYPES OF NOISE

- **Steady or Continuous Noise:** It is uninterrupted noise that varies less than 5 dB-A during the period of measurement eg Noise from house hold fan , boiler in a power house, lathes, diesel engine ,grinder etc
- **Impulsive or Impact noise :**When source causes vibration for a short time eg firing from the gun or hammer
- **Intermittent/Fluctuating Noise:** Large workshop number of machines are in operation, noise levels varies from time to time or dentists drilling

SELECTION OF INSTRUMENT, SAMPLING DURATION AND SCALE

- To measure noise level, the most extensively used instrument is a Sound Level Meter (SLM) which commonly is known as a noise meter.
- After selection of instrument, selection of sampling duration and scale is must. With these it is also necessary to consider the parameters which we need to measure like; L_{max} , L_{min} , L_{eq} , etc.

SOUND LEVEL METER

- Sound level meter SLM consist of a microphone, electric circuit and digital display. Microphone detects and reads minimal air pressure changes and converts them into electric signals.
- These signals are processed by electric circuits and converted into decibels. SLM can read noise level for one location at a time.
- While measuring noise levels it is held in an arm's length at the ear height. It does not matter whether microphone is point towards source.
- Sound level monitor must be calibrated before and after use.
- Every sound level meter has two modes and those are SLOW and FAST.
- It is the response rate which SLM averages before showing on screen.
- For workplace noise level monitoring it should be taken as SLOW.



Sound Level Meter

INFORMATION REQUIRED FOR NOISE SURVEY/MONITORING

- Location and Nature of work (Sketch of the measurement site, including size of the room, machine dimensions etc.)
- Nature of work process and task
- No of workers working in noisy area
- Current control including protection devices (e.g. Personal Protection devices like ear plugs or ear muffs)
- Community noise monitoring is required? If yes, Status of Community monitoring, whether higher than permissible level?
- Type of sound level meter
- Operating condition during the measurement and job description
- Results of the noise assessment
- Date of assessment or monitoring
- Name and signature of the person conducting the monitoring.

NOISE LEVEL MEASUREMENT PROCEDURE

- Must be check the battery full before goes onside monitoring.
- Switch ON of equipment and wait for one minute.
- SLM has two modes of operation selection via the menu key.
- Two mode:(1) In continuous Mode (2) In Recording Mode

In continuous Mode: For only onsite observation.

In Recording Mode: For recording the data in sound level meter.

- Select the appropriate mode of sound level.
- Set the slow or fast time as per site condition. Slow time is applicable for normal measurement. Fast time is applicable for specific measurement of noise like moving train, heavy traffic etc.
- Adequate Distance (1 to 3 meter) maintain between source and equipment.
- Take reading for at least one minute at one location. Number of reading can take for one minute intervals.
- Monitoring period should be decided in such a manner that one reading is available after one minute of monitoring at one location.
- When we change the location, off the machine and again Switch ON of equipment.

INSTRUMENT PLACEMENT

Many factors need to be taken into account when measuring because sound levels vary at different heights above ground level. They will also vary depending on the distance between the measurement point and facades and obstacles. These are some important factors for doing monitoring:

- a. Away from facades
- b. Away from obstacles
- c. With the microphone 1.2 - 1.5 meter above ground level
- d. Monitoring inside Industrial facility distance from source shall be 1.5 meter

MONITORING LOCATIONS

The locations for monitoring to assess the ambient noise levels shall be mix up of all zones i.e. Residential, Commercial and Industrial to find the variations with different zones. The monitoring schedule carried out on working days and weekend to differentiate the noise levels between normal days to weekend days. The Sampling locations should be sufficient enough provide representative samples for the project.

COMPARISON WITH NOISE STANDARDS

Once we get the monitoring records then we can compare them with standards to understand if noise levels are exceeding the standards and if they are then by how much. In India, the standards extensively used are prescribed by Central Pollution Control Board (CPCB).

NOISE LEVEL MEASUREMENT STANDARDS

- Limits or noise level standards defined by pollution control board during Day time for Residential Noise < 55 dB and Limits during Night time for Residential Noise < 45 dB
- In industry, there are of two types Sound Monitoring & testing or Noise Level Measurement: (A) Ambient Noise level Monitoring & (B) In-plant Noise level Monitoring

A) Ambient Noise level standards

Ambient Noise level Monitoring or Noise pollution Measurement within industrial zone at ambient conditions.(e.g. Near Main Gate, Near Canteen, Near Manufacturing plant etc.) As per Central Pollution control Board (as per Factory act 1948).

Limits or acceptable noise level during Day time for Ambient Noise < 75 dB and Limits during Night time for Ambient Noise < 70 dB.

B) In-plant Noise level Standards

In-plant Noise level means Sound level measurement allocated in the plant. As per Central Pollution control Board (as per Factory act 1948) Limits for In-plant Noise level < 90 dB.

- The below table shows the Noise level Monitoring standards prescribed by Central Pollution Control Board (CPCB). Noise Pollution (Regulation & Control) Rules 2000 under the provision of the Environment (Protection) Act, 1986. These rules provides standards in respect of noise for different areas/zones of a city/town

Area code	Category of area/Zone	Limits in db (A)	
		Day Time	Night Time
A	Industrial area	75	70
B	Commercial area	65	55
C	Residential area	55	45
D	Silence zone	50	40

Day time : 6:00 AM to 10:00 PM

Night time : 10:00 PM to 6:00 AM