ADICHUNCHANAGIRI INSTITUTE OF TECHNOLOGY
CHIKKAMAGALURU-577102

Department of Civil Engineering

ENVIRONMENTAL ENGINEERING LABORATORY

15CVL76

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The Program Educational Objectives (PEOs)

1. Able to apply the knowledge of Engineering to solve construction related problems pursue higher education and involve in research activities.
2. Able to Plan, design and execute the societal applications of Civil engineering.
3. To Involve effectively as a member or as a leader towards achieving goals in Civil engineering projects.
4. To Engage in professional consultancy and continuous learning to accomplish Professional growth

Program Outcomes (PO's)

The graduates of the Civil Engineering Department will have the ability of

1. **Engineering knowledge**: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization t o the solution of complex engineering problems.
2. **Problem analysis**: Identify, formulate, research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
3. **Design/development of solutions**: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
4. **Conduct investigations of complex problems**: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
5. **Modern tool usage**: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations
6. **The engineer and society**: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.

7. **Environment and sustainability**: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.

8. **Ethics**: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.

9. **Individual and team work**: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.

10. **Communication**: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.

11. **Project management and finance**: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.

12. **Life-long learning:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change

Program Specific Outcomes (PSO's)

By the completion of Civil engineering program, graduates are able to

1. Utilize the Civil engineering knowledge and problem analysis skills to conceptualize, develop, and execute the civil engineering projects.
2. Deploying quality Civil Engineers to work towards societal needs to achieve environmental and sustainable development

COURSE OUTCOMES:

CO1: To understand the importance of sample collection and analysis.

CO2: Determine chemical, physical and biological characteristics of water and wastewater.

CO3: Determine optimum dosage of coagulant, Residual chlorine and available chlorine

CO4: Determination of Nitrates, Iron and manganese by Spectrophotometer.

DOs and DON'Ts in the Laboratory

1. Do thoroughly clean the glassware before and after use.

2. Do handle the glassware carefully.

3. Do not handle chemicals with bare hands.

4. Do not blow out the last drop from the pipette. When the liquid has drained out completely, touch the tip of the pipette to the inner surface of the vessel.

5. Do not add water to acids. Do always add acid to water.

6. Do use large volumes of water, when a person is splashed with acid to prevent serious burns.

7. Do weigh the articles in a balance only at room temperature.

8. . Do use different pipette for different reagents

9. Do not pipette out acids and other toxic reagents by mouth.

10. Do read the level of the curve (meniscus), in all volumetric glassware, with the eye at approximately the same level as the curve of solution.

General Information

In water and wastewater analysis, the results are usually reported in terms of mg/L of some particular ion, element or compound. It is most convenient to have the standard titrating agent of such strength, that 1mL is equivalent to 1mg of material being measured. Thus 1 litre of the standard solution is usually equivalent to 1g of the standard substance.

Normality

The desired normality of the titrant is obtained by the relationship of 1 to the equivalent weight of the measured material. Thus normality of acid solution to measure ammonia, ammonia nitrogen, and alkalinity as $CaCO_3$

Ammonia — 1/eq. wt. = 1/17 = N/17 = 0.0588N

Ammonia N — 1/eq. wt. = 1/14 = N/14 = 0.0715N

Total $\underline{Solids} = \underline{W_2-W_1}x10^6$ mg/L

      ml of sample

Total $\underline{Solids} = \underline{W_2-W_1}x10^6$ mg/L

      ml of sample

The normality of basic solution to measure mineral acidity as $CaCO_3$ is:

Acidity — 1/eq. wt. = 1/50 = N/50 = 0.020N

The normality of silver nitrate to measure chloride and sodium chloride is:

Chloride — 1/eq. wt. = 1/35.45 = N/35.45 = 0.0282N

Sodium chloride — 1/eq. wt. = 1/58.44 = N/58.44 = 0.071N

Thus the substance measured is calculated as follows:

= ml of titrant used × 1,000 mg / L

      ml of sample

Most materials subjected to the analysis of water and wastewater fall in the realm of dilute solutions i.e., a few mg in a litre. So the results are normally expressed in mg/L or ppm. Parts per million (ppm) is a weight ratio; but mg/L is a weight by volume ratio. The relationship is given as follows:

      ppm = mg / L

If concentrations are less than 0.1 mg /L, express them in µg/L (micrograms per litre).

If concentrations are more than 10,000 mg/L, they are expressed in percentages

Lab Safety:

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.

Lab Etiquette:

Return all chemicals and supplies to the proper location after use. It is necessary to take chemicals from reagent bottles, pour out slightly more than the amount of chemical needed into a clean beaker. Never pour a chemical back into a reagent bottle. Clean up for the next person. At the conclusion of each work period, all used glassware must be cleaned and set to drain. Remove label tape, scrub inside of glassware with water and laboratory detergent, rinse with tap water, rinse with distilled water, and place cleaned glassware on a rack to dry. No experiment is complete until the laboratory is cleaned.

Procedure for cleaning of glassware in laboratory

Glassware cleaning

1. Clean the equipment thoroughly with soap and water for basic cleaning. You may need to use a wire brush to remove some residue. Detergent using bottle brushes and scouring pads can be used as needed.

2. After cleaning, rinse the glassware with running tap water. When test tubes, graduates, flasks and similar containers are rinsed with tap water, allow the water to run into and over them for a short time, then partly fill each piece with water.

3. Thoroughly shake and empty at least six times and ensure that all soap residue is removed.

Note:

• Do not use cleaning brushes that are so worn that the spine hits the glass. Serious scratches may result. Scratched glass is more prone to break during experiments. Any mark in the uniform surface of glassware is a potential breaking point, especially when the piece is heated. Do not allow acid to come into contact with a piece of glassware before the detergent (or soap) is thoroughly removed. If this happens, a film of grease may be formed.

• To prevent breakage when rinsing or washing pipettes, cylinders or burettes, be careful not to let tips hit the sink or the water tap.

Sterilizing Contaminated Glassware

• Autoclave glassware or sterilize it in large steam ovens or similar apparatus. If viruses or spore bearing bacteria are present, autoclaving is absolutely necessary.

Handling and Storing

• Protect clean glassware from dust. This is done best by plugging with cotton, corking, taping a heavy piece of paper over the mouth or placing the glassware in a dust-free cabinet.

• Store glassware in specially designed racks. Avoid breakage by keeping pieces separated.

Meaning of Water Quality Analysis

Water quality standards are put in place to ensure the efficient use of water for a designated purpose. Water quality analysis is to measure the required parameters of water, following standard methods, to check whether they are in accordance with the standard.

Requirement of Water Quality Analysis

Water quality analysis is required mainly for monitoring purpose. Some importance of such assessment includes: **1**. To check whether the water quality is in compliance with the standards, and hence, suitable or not for the designated use. **2**. To monitor the efficiency of a system, working for water quality maintenance. **3**. To check whether up gradation / change of an existing system is required and to decide what changes should take place. **4**. To monitor whether water quality is in compliance with rules and regulations.

Sampling of Water for Analysis

A common cause of error in water quality analysis is improper sampling. The results of a water quality analysis of a sample show only what is in the sample. For the results to be meaningful, the sample must be representative i.e., it must contain essentially the same constituents as the body of water from which it was taken. The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled. The complexity of water quality as a subject is reflected in the many types of measurements of water quality indicators. The most accurate measurements of water quality are made on-site, because water exists in equilibrium with its surroundings. Measurements commonly made on-site and in direct contact with the water source in question include temperature,' pH, dissolved oxygen, electric conductivity, etc. More complex measurements are often made in a laboratory requiring a water sample to be collected, preserved, transported, and analyzed at another location.

Requirements for Sampling

• Meet the requirements of the sampling program. • Handle the sample carefully so that it does not deteriorate or become contaminated or compromised before it is analyzed. • Ensure sampling all equipment are clean and quality assured before use. • Use sample containers that are clean and free of contaminants. • Rinse the bag/bottle at least twice with the sample water prior to filling and closing. • Fill bag/bottle as full as possible. Half-filling leaves more room for oxygen which will promote degradation of your sample. • If sampling a body of running water, point the mouth of the bag upstream and your hands downstream to avoid contamination. • If sampling from a water faucet, run the faucet for 1 minute before obtaining a sample. • Make records of every sample collected and identify every bottle e.g., take notes and photographs, fill out tags, etc. • Place the sample into appropriate, labeled containers. • All samples must be preserved as soon as practically possible.

Sample Collection bottles, Size and Materials

The methods that will be followed will determine the type of bottles used. For example, samples for metals' analyses are usually collected in plastic bottles, while analyses for volatile organics and pesticides are collected in glass containers. Bottles used to collect samples for bacteria should be sterilized. Certain analysis like volatile organics and radon require vials that are to be filled leaving no head space, which keeps these analytes

dissolved in the water, preventing them from escaping into the air. Additionally, some analyses require samples to be collected in amber colored bottles. These darker bottles are for analytes that break-down in sunlight, which helps keep these contaminants from breaking down while in transit to the laboratory for analysis. The size of the container is important to ensure enough samples to run the analysis needed.

Water Sampling Techniques: Water sampling can be done in any of the following three methods depending on test requirements: • Grab sampling • Composite sampling • integrated sampling

Grab sampling: Grab Samples are samples collected at a particular time and space. They represent the composition at that time and place. When a source is known to vary in time e.g.in case of waste effluents, grab samples collected at suitable time intervals and analyzed separately.

Composite sampling: Composite samples are a mixture of grab samples collected at one sampling point at different times. The composite samples are useful for observing values. Individual samples are collected in wide mouth bottles every hour and mixed in volume proportional to the flow or by using specially designed automatic sampling devices.

Advantages of Composite Samples:

- reduced costs of analyzing a large number of samples.

- More representative samples of varied conditions.

- Larger sample sizes when amounts of test samples are limited.

Disadvantages of Composite Samples:

- Loss of analytic relationships in individual samples.

- Potential dilution of any parameter below detection levels.

- increased potential analytical interferences.

 - increased possibility of analytic interactions.

Integrated sampling: Integrated samples are a mixture of grab samples collected from different points simultaneously and mixed in equal volumes.

Surface Water Sampling Techniques:

When the water source is accessible

Rinse the sampling vessel with water on site 3-4 times. Care must be taken to avoid contaminating water to be sampled during rinsing. - Submerge the sampling vessel gently, fill it with the water sample and close it tightly. If the collected water sample may be frozen, leave some space for expansion equivalent to about 10% of the sampling vessel.

When the water source is inaccessible-

A rope 'attached to the bucket are often used. Scoops with adjustable shafts are convenient. Items made of synthetic resins such as polypropylene can also be used.

Ground Water (from well) Sampling Techniques

A bailer in is a hollow tube used to retrieve groundwater samples from monitoring wells. Bailers are tied to a piece of rope or a piece of wire and lowered into the water column. Once lowered, the bailer uses a simple ball check valve to seal at the bottom in order to pull up a sample of the groundwater table.

CONTENTS

EXPERIMENT – 1: Determination of Alkalinity, Acidity and pH

a) ALKALINITY

Aim: To determine the Alkalinity of the given water sample.

Apparatus:
- 250 ml conical flasks
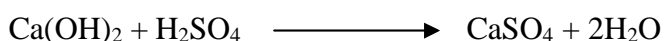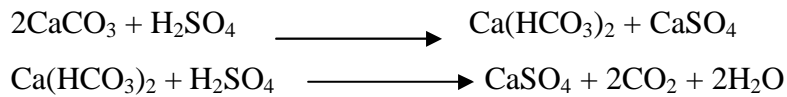- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Reagents:
- 0.02N or N/50 Sulphuric acid (H_2SO_4)
- Methyl orange indicator
- Phenolphthalein indicator.

Theory:

Alkalinity or acid neutralization is the capacity of water to neutralize hydrogen ions. In natural water, alkalinity is mainly due to the presence of Bicarbonates, carbonates and hydroxides of calcium, magnesium, sodium and potassium. However, hydroxide salts are present in low levels (Sawyer et. al., 1994). Bicarbonates epitomize the major form of alkalinity due to the action of carbon dioxide upon basic materials in soil. Highly alkaline waters are unsuitable for domestic, industrial and agricultural purposes. Hence determination of alkalinity is important and is measured volumetrically against 0.02 N H_2SO_4). Other salts of weak acids such as borates, silicates, phosphates and organic salts produce alkalinity in very small amounts and hence ignored.

Caustic alkalinity or hydroxyl alkalinity is due to the presence of hydroxides. This raises the pH of water considerably. During titration with an acid all the hydroxide alkalinity is completely neutralized by the time pH decrease to 10. Further all the carbonates get converted to bicarbonates by the time pH reaches 8.3. Bicarbonates exist up to a pH of 4.5. For this reason phenolphthalein indicator is used for pH 8.3 and Methyl orange indicator for pH 4.5. Hydroxyl alkalinity and bicarbonate alkalinity cannot exist together for a sample.

$$Ca(OH)_2 + H_2SO_4 \longrightarrow CaSO_4 + 2H_2O$$

$$2CaCO_3 + H_2SO_4 \longrightarrow Ca(HCO_3)_2 + CaSO_4$$

$$Ca(HCO_3)_2 + H_2SO_4 \longrightarrow CaSO_4 + 2CO_2 + 2H_2O$$

Alkalinity is measured in two forms:

i) Partial alkalinity or Phenolphthalein alkalinity

ii) Total alkalinity or Methyl orange alkalinity.

Hydroxide, Carbonate and Bicarbonate alkalinity for the given water sample can be computed knowing the values of Partial and total alkalinity.

Table 1: Alkalinity Relationship

SL NO.	Alkalinity Relationship	Hydroxide or caustic alkalinity	Carbonate alkalinity	Bicarbonate alkalinity
1	P=0	0	0	T
2	$P<^1/_2 T$	0	2P	T-2P
3	$P=^1/_2 T$	0	2P	0
4	$P>^1/_2 T$	2P-T	2(T-P)	0
5	P=T	T	0	0

P = Partial or phenolphthalein alkalinity
T = Total alkalinity

Procedure:

a) Partial or Phenolphthalein Alkalinity

1. Take 100ml of sample in a clean conical flask.
2. Add 2-3 drops of phenolphthalein indicator solution and shake well the contents in the flask.
3. Note the initial burette reading (R_1).
4. If no pink color is seen, phenolphthalein alkalinity is nil in the sample (P = 0).
5. If pink color appears in the sample, titrate with 0.02 N H_2SO_4 reagents till the end point is reached to colorless.
6. Note the final burette reading (R_2).

Tabular column

SL. No	Sample	Burette Reading			Average Volume of H_2SO_4
		Initial Reading (IR)	Final Reading (FR)	Volume of 0.02 N H_2SO_4 consumed	
1					
2					
3					
4					
5					
6					

Calculations:

Phenolphthalein Alkalinity as mg/L $CaCO_3$ =

$$\frac{V_1 \times \text{Equivalent weight of } CaCO_3 \times \text{Normality} \times 1000}{\text{ml of sample taken}}$$

Where V_1 = Volume of 0.02N or N/50 H_2SO_4 consumed

N = Normality of H_2SO_4

50 = Equivalent Weight of $CaCO_3$

b) Total or Methyl orange Alkalinity:

1. Add 2-3 drops of methyl orange indicator solution to the solution in which phenolphthalein alkalinity has been determined.

2. Shake well the contents in the conical flask.

3. It will turn to yellow color.

4. Titrate with 0.02 N H_2SO_4, till the orange red color appears.

5. Note down the volume of the reagent consumed (V_2).

Tabular column 2

SL. No	Sample taken	Burette Reading			Average Volume of H_2SO_4
		Initial reading (IR)	Final reading (FR)	Volume of H_2SO_4 consumed	
1					
2					
3					

Calculations:

Total Alkalinity as mg/L $CaCO_3$=

$$\frac{(V_1 + V_2) \times \text{Equivalent of } CaCO_3 \times \text{Normality} \times 1000}{\text{ml of sample taken}}$$

Where V_2= ml of H_2SO_4 titrant

consumed; N = Normality of

H_2SO_4 (1/50); 50 = Equivalent

Weight of $CaCO_3$

Results and Discussion:

Calculate Hydroxide, Carbonate and Bicarbonate Alkalinities using Alkalinity Relationship table

Sl. No.	Type of Alkalinity	Result as mg/L $CaCO_3$
1	Phenolphthalein alkalinity	
2	Total alkalinity	
3	Hydroxide or caustic alkalinity	
4	Carbonate alkalinity	
5	Bicarbonate alkalinity	

Environmental Significance:

a) Water:

Alkalinity of water has little public health significance. Highly alkaline, waters are usually unpalatable. Some amount of alkalinity is however desirable as it provides essential minerals and prevents corrosion. Caustic alkalinity is not desirable. Alkalinity reacts with coagulants to produce flock particles, which aid in the removal of fine colloidal particles from water. Most of the minerals producing alkalinity also cause hardness of water.

The knowledge of different types of alkalinity is needed in water softening processes and in boiler water analysis.

b) Sewage Alkalinity:

Alkalinity of sewage:

Sewage when fresh is slightly alkaline. Due to biological reactions and consequent production of organic acids, sewage becomes septic and acidic. After completion of biological reactions, sewage once again becomes alkaline.

Alkalinity of sewage is an index of its age and degree of decomposition of organic content. Alkalinity of wastewater is important where chemical treatment is used and where ammonia is to be removed by air stripping.

Standard Unit of Alkalinity: 'Alkalinity is expressed as mg/L equivalent amount of $CaCO_3$.

Permissible limit: The standard permissible limit for alkalinity is 250 mg/L for domestic usage.

Removal Techniques: removal of alkalinity is not usually needed. When required to remove, caustic alkalinity is removed done by neutralizing with an acid.

b) ACIDITY

Aim: To determine the acidity of the given water sample.

Apparatus:
- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Reagents:
- NaOH [0.02N]
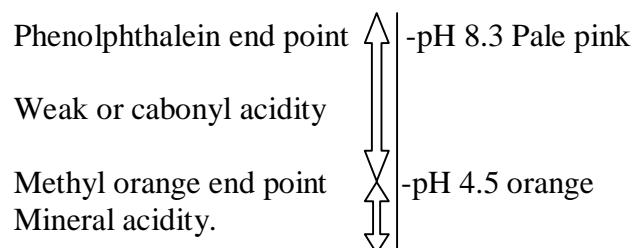- Methyl orange indicator
- Phenolphthalein indicator.

Theory:

Most of the natural waters are buffered by a carbon dioxide – carbonate system since carbon dioxide is a natural component of all natural waters. This causes water to be slightly acidic and acidity can be defined as the capacity of water to neutralize hydroxyl ions.

Acidity mainly caused due to the presence of mineral acids and free carbon dioxide in water. Carbon dioxide can also be produced in water through biological oxidation of organic matter, especially in polluted water. Acidity (both carbon dioxide and mineral) is estimated through titration with a base 0.02 N sodium hydroxide (NaOH).

Carbon dioxide and organic acids produce weak acidity and are natural components of water. Mineral acidity is caused due to industrial wastewater contamination of water.

During titration with a standard alkali like NaOH, the mineral acidity, which lowers the pH to very low values, is neutralized first. At a pH of 4.5, mineral acidity will have been completely neutralized. Beyond pH 4.5 carbon dioxide acidity will be present up to a pH of 8.3. Hence two indicators Methyl orange for pH 4.5 and Phenolphthalein for pH of 8.3 are used.

Phenolphthalein end point ⇕ -pH 8.3 Pale pink

Weak or cabonyl acidity

Methyl orange end point ⇕ -pH 4.5 orange
Mineral acidity.

pH scale for end point

Procedure:

a) **Methyl orange acidity Mineral acidity**

1. Take 100ml of sample in a clean conical flask.

2. Add 2 to 3 drops of methyl orange indicator and color of the sample changes into orange red.

3. Titrate with 0.02 N NaOH till the end point of yellow color appears.

4. Note down the volume of the burette reading (V_1).

Tabular Column 1

Sl. No	Sample taken	Burette Reading			Average Volume of NaOH
		Initial Reading (IR)	Final Reading (FR)	Volume of NaOH consumed	
1					
2					
3					

Calculations: Mineral acidity as mg/L $CaCO_3$ =

$$\frac{V_1 \times \text{Equivalent. Weight of } CaCO_3 \times \text{Normality x } 1000}{\text{ml of sample taken}}$$

Where V_1 = Volume of NaOH titrant consumed

N= normality of NaOH;

50= Eq.wt of $CaCO_3$

1 ml of 0.02 N NaOH is equivalent to 1 mg $CaCO_3$

b) **Phenolphthalein acidity or Total Acidity:**

1. Pipette out 50 ml sample into a 250 ml conical flask.
2. Add 2 to 3 drops of Phenolphthalein indicator solution.
3. Note down the initial burette reading.
4. Titrate against 0.02 N sodium hydroxide until the colorless sample changes to pink color.
5. Note down the volume of NaOH consumed (V_2).

Tabular column 2

Sl. No	Sample taken	Burette Reading			Average volume of NaOH
		Initial reading (IR)	Final Reading (FR)	Volume of NaOH consumed	
1					
2					
3					

Phenolphthalein acidity as mg/L $CaCO_3$=

$$\frac{V_2 \times \text{Equivalent weight of } CaCO_3 \times \text{Normality} \times 1000}{\text{ml of sample taken}}$$

Where V_2 = Volume of NaOH titrant consumed

N = normality of NaOH,

Acidities determined using these indicators are known as

   i) Methyl orange acidity or Partial acidity or Mineral acidity

   ii) Phenolphthalein acidity or Total acidity

Carbon dioxide acidity is the difference of total acidity and mineral acidity.

CO_2 acidity = Total acidity- Partial acidity.

Environmental Significance:

          Acidity is of little concern from public health viewpoint. Carbon dioxide acidity is absolutely harmless. Mineral acidity makes the water so unpalatable that problems concerned with human consumption are nonexistent. Acidity is significant because it contributes to corrosiveness of water. Acidity is of concern because of its corrosiveness.

Standard unit of acidity:

Acidity is expressed, as mg/L equivalent amount of $CaCO_3$ i.e. 1 unit of acidity is that required neutralize 1 mg/L of alkalinity as $CaCO_3$.

Permissible limit:

There is no prescribed limit for acidity. However mineral acidity should be absent in public water supplies, as it is more serious.

Removal techniques:

CO_2 acidity is removed by aeration and mineral acidity is removed by neutralizing with an alkali.

Results and discussion:

c) pH

Aim: To determine the pH of the given sample using pH paper, Universal indicator and pH meter

Apparatus:
- Test tube
- Glass rod and Beaker
- pH meter

Theory:

pH indicates the intensity of acid or alkaline condition of a solution. It is a way of expressing the hydrogen ion concentration, or more precisely, the hydrogen ion activity. pH of most natural waters lie within the range of 4 to 9.

Acid range	Alkaline range	Neutral pH
0-7	7-14	7
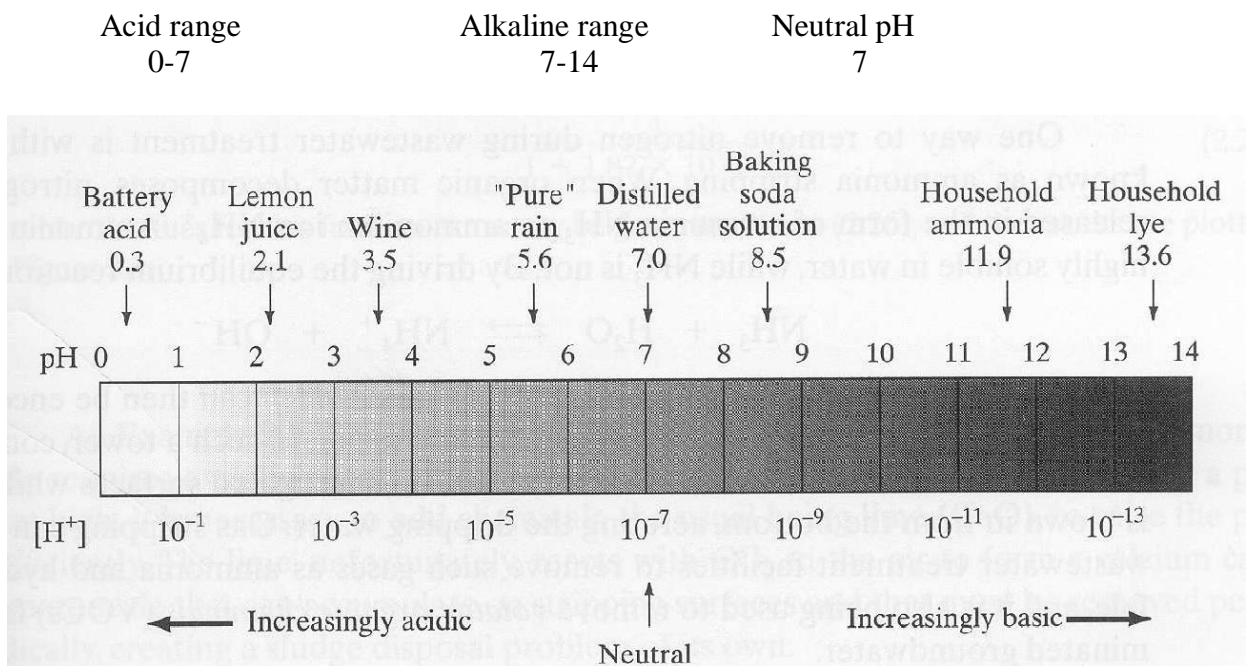
Figure. pH scale

Procedure:

1. Using pH papers: Dip a strip of pH paper and compare the change in color of paper with standard color chart to get the pH value.

2. Using Universal indicator: Take 10ml sample in a test tube, add 0.2ml (2 drops) of

universal indicator shake the test tube compare the color developed with standard chart to get the pH value.

3. Electrometric method: Using glass electrode

Principle: A glass electrode in combination with a calomel electrode is commonly used.

The combination of these two electrodes dipped in the sample, which acts as an electrolyte act as an electric cell. The current, which flows through the cell, is dependent upon the pH of the sample. V

Description:

The apparatus consists of two electrodes with a calibrated voltmeter which reads either pH or mule volts of current directly. The pH meter consists of a calibration correction knob, a scale selection knob for selecting acidic, alkaline or neutral range of pH scale. Temperature of sample brings about variation of electrode potential. This effect can be corrected by a temperature correction know. The effect of temperature on sample pH is considered by recording the temperature also along with pH.

Procedure:

Standardize the instrument against a buffer solution of known pH almost that of the sample. Check the linearity of electrode response against one or more additional buffer solutions. Every time before the electrode is put into a different electrolyte (sample) rinse the electrode with distilled water and wipe it dry was using an absorbent tissue.

Accuracy: The glass electrode is immune to the interference from colour, turbidity, free chlorine, Oxidants or reducing agents and also from a high saline content.

Results and discussion:

Sl. No	Sample taken	pH			Average pH
		pH Paper	pH Indiactor	Potentiometer	
1					
2					
3					

Significance:

Addition of an acid to water increases the hydrogen ion concentration in water and hence the pH will be less than 7. Addition of a base will increase the hydroxyl ions and reduce the hydrogen ion concentration. Hence the pH value will increase. Several Processes like softening, coagulation, adsorption, gas transfer are pH specific. Changes in pH will cause dissociation of ions and hence will influence the chemical reaction. When the pH has to be maintained in a chemical process, buffer solutions (solutions which resists the changes in pH) will be used. Some chemicals also change their colour due to release of specific ions at specific pH values and are used as indicators. Very low and very high pH values are no suitable for biological metabolism and bacterial growth.

Environmental significance:

Acidity and alkalinity express the total reserve or buffering capacity of a sample, pH value represents instantaneous hydrogen ion activity

In the field of water supplies pH must be considered in Chemical coagulation, disinfection, water softening and corrosion control. For public water supplies pH must be kept as close to 7 as possible. Acidic waters cause corrosion & tuberculation. Alkaline water may produce incrustation sediment deposits, difficulty in chlorination. Permissible pH values for public supplies may range between 7 and 8.5 (acceptable) and 6.5 to 9.2 (rejection)

In waste water systems pH plays an important role in biological treatment (aerobic and anaerobic). Chemical processes for coagulating wastewater, dewatering sludge's or oxidizing substances like cyanide etc require proper control of pH.

Measurement of pH: pH can be measured either by colorimetry OR electrometry.

Colorimetric methods are cheaper but affected by interference due to the presence of colour, turbidity, Salinity, free chlorine and oxidants and reducing agents, Colorimetric methods are suitable only for rough determination of pH.

EXPERIMENT – 2: Determination of Calcium, magnesium and Total Hardness

a) Total Hardness

Aim: To determine Total Hardness content in the given water sample.

Apparatus:

- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Theory:

Water hardness is the capacity of water to react with soap to produce sufficient lather. Hardness of water is not a specific element but a variable accounted by a complex mixture of cations and anions. Calcium, magnesium, strontium, ferrous ion and manganous ions are the chief harness causing divalent cations. These cations, plus the significant anions with which they area associated are shown in the following table in the order of their relative abundance in natural waters (Sawyer et. al., 1994).

Relative abundance of cations and anions causing hardness

Cations causing hardness	Anions
Ca^{2+}	HCo_3^-
Mg^{2+}	So_4^{2-}
Fe^{2+}	No_3^-
Mn^{2+}	SiO_3^{2-}

Hard waters are as satisfactory as soft waters from human consumption point of view. Due to adverse action with soap their use for cleansing purpose, hard water use is generally avoided or used after treatment. Hard water lead to scale formation in heaters, boilers and other units in which water temperature is increased materially and hence are unfit for industrial purposes.

Water is classified from soft to very hard depending on the amount of hardness quantified as $CaCO_3$ in milligrams per litre of the sample as shown below.

Classification	Hardness as CaCO₃ in mg/l
Soft	0-75
Moderately hard	75-150
Hard	150-300
Very hard	> 300

Total hardness can be classified as temporary hardness and permanent hardness.

Temporary Hardness: Temporary hardness is caused by bicarbonates of calcium and magnesium ions. This is removed simply by boiling the water.

By Boiling of water:

$$Ca(HCO_3)_2 \longrightarrow CaCO_3 + H_2O + CO_2$$

$$Mg(HCO_3)_2 \longrightarrow MgCO_3 + H_2O + CO_2$$
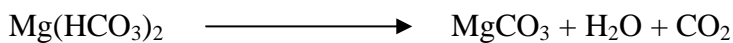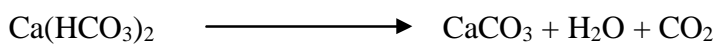
Since these bicarbonates contribute towards the alkalinity of water. The temporary hardness can be conveniently removed or determined by estimating the alkalinity of water sample before and after boiling by titration.

Permanent Hardness:

The permanent hardness is due to the dissolved sulphates, chlorides and nitrates of calcium and magnesium and it cannot be removed by boiling. The permanent hardness can be removed by Lime-Soda process and Zeolite process.

Principle:

When EDTA is added to water containing both Ca& Mg, it combines first with Ca, Mg is precipitated as hydroxide by raising the pH to 12-13 using NaOH. After precipitating Magnesium, remaining Calcium is directly determined by titrating with standard EDTA. Ca and Mg ions form stable complex salt with EDTA.

EDTA forms a more stable complex with M^{++} (hardness ions) and frees Erichrome black T indicator and the wine red colour changes to blue color. This indicates the end of titration. At pH greater than 10, $Ca(OH)_2$ and $Mg(OH)_2$ are precipitated. This pH is achieved by adding ammonia buffer to the sample.

Unit of hardness:

Hardness is expressed as mg/L as CaCO₃. Sometimes it is expressed as degree of

hardness 1 degree of hardness is equivalent to 14.25 mg/L as $CaCO_3$.

Procedure:

a) Total Hardness

- Take 100 ml of sample in a conical flask.
- Add 1 ml of Ammonia buffer solution to the sample.
- Add a pinch of Erichrome Black – T indicator, the sample will turn to wine red color.
- Titrate it with 0.01 M (molar) EDTA solution until wine red color changes to blue color.

b) Permanent Hardness

Follow the same above procedure for boiled and cooled water sample.

c) Temporary Hardness = (Total hardness – Permanent hardness)

Calculations:

a) Total Hardness

Tabular column

Sl. No	Sample taken	Burette Reading			Average volume of EDTA
		Initial reading (IR)	Final Reading (FR)	Volume of EDTA consumed	
1					
2					
3					

Total hardness (mg/l as $CaCO_3$) = $\dfrac{V \times B \times 1000}{ml \ of \ sample \ taken}$

Where, V = Volume of reagent EDTA consumed

B = mg of $CaCO_3$ equivalent to 1 ml of EDTA (B is taken as 1)

b) Permanent Hardness (Boiled and Cooled water)

Sl. No	Sample taken	Burette Reading			Average volume of EDTA
		Initial reading (IR)	Final Reading (FR)	Volume of EDTA consumed	
1					
2					
3					

Permanent hardness (mg/l as $CaCO_3$) =

$$\frac{V \times B \times 1000}{\text{ml of sample taken}}$$

Where, V = Volume of reagent EDTA consumed

B = mg of $CaCO_3$ equivalent to 1 ml of EDTA (B is taken as 1)

c) Temporary Hardness = Total Hardness – Permanent hardness

Environmental Significance:

Hard waters are as satisfactory for human consumption as soft waters. Small amount of hardness imparts taste to water and adds essential minerals such as Ca and Mg. It also protects against corrosion as very soft waters are corrosive. Excessive hardness causes consumption of large amounts of soap for washing. It imparts saline taste to water. It causes boiler scale formation and incrustation of pipes. Hardness of water can be reduced by (i) Boiling (ii) Lime soda process (iii) Zeolite or Base Exchange process (iv) Ion exchange process.

Removal techniques:

Softening techniques such as boiling, lime soda process, Zeolite process and Ion exchange process may be used for removal of excess Calcium and Magnesium.

b) Calcium Hardness

Aim: To determine Calcium Hardness content in the given water sample.

Apparatus:

- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Theory:

Calcium is a major constituent of most igneous, metamorphic and sedimentary rocks and is also present in the form of adsorbed ions on negatively charged mineral surfaces in soils and rocks. Calcium and magnesium cause by far the greatest portion of the hardness occurring in natural waters. Calcium does not have any psychological effects except for their action with soap and encrustation in water supply structures, boilers and vessels used for storing water.

Procedure:

1. Take 100 ml of sample in a conical flask,

2. Add 2 ml of 1N (or saturated) NaOH solution.

3. Mix well the contents in the flask by swirling.

4. Add a pinch of Patton and Reeder's indicator, and sample will turns to pink color.

5. Titrate it with 0.01M Ethelene diamine tetr acitic acid (EDTA) solution until pink color turns to purple color.

6. Note down the burette reading.

Calculations:
Tabular Column: Calcium hardness

Sl. No	Sample taken	Burette Reading			Average volume of EDTA
		Initial reading (IR)	Final Reading (FR)	Volume of EDTA consumed	
1					
2					
3					

$$\text{Calcium hardness (mg/l as } CaCO_3) = \frac{V \times B \times 1000}{\text{ml of sample taken}}$$

Where, V = Volume of reagent EDTA consumed

B = mg of $CaCO_3$ equivalent to 1 ml of EDTA (B is taken as 1)

Calcium hardness (mg/l as Ca) = Calcium hardness (mg/l as $CaCO_3$) × 0.40

c) Magnesium hardness

Aim: To determine Magnesium Hardness content in the given water sample.

Theory:

Magnesium is a significant constituent of basic igneous rocks like dunites, pyroxenites and amphibolites, volcanic rocks such as basalts, metamorphic rocks, for example talc, tremolite, schists, and of sedimentary rocks such as dolomite and tainted limestone, which also contains some amount of magnesium carbonate. Dolomite, olivine, augite, biotite, hornblende, serpentine and talc are some of the main magnesium bearing rock forming minerals. Although in the igneous and metamorphic rocks, magnesium occurs in the form of insoluble silicates, weathering breaks them down into more soluble carbonates, clay minerals and silica. In the presence of carbonic acid in water, magnesium carbonate is converted into more soluble bicarbonates.

$$MgCO_3 + CO_2 + H_2O \longleftrightarrow Mg(HCO_3)_2$$

In normal atmospheric conditions, the solubility of magnesium carbonate in water in presence of carbon dioxide is nearly ten times that of calcium carbonate. calcium and magnesium cause by far the greatest portion of the hardness occurring in natural water. Magnesium does not have any psychological effects except for their action with soap and encrustation in water supply structures, boilers and vessels used for storing water.

Calculations:

Magnesium hardness of the sample is calculated as follows;

Magnesium hardness in mg/l as $CaCO_3$ =

(Total Hardness in mg/l as $CaCo_3$ ⎯ Calcium hardness in mg/l as $CaCO_3$)

Magnesium in mg/l as Mg =

(Total Hardness in mg/l as $CaCO_3$ ⎯ Calcium hardness in mg/l as $CaCO_3$) × 0.244

Results: Express the results as magnesium hardness in mg/l as CaCO$_3$ or magnesium (as Mg) in mg/l

Environmental Significance:

Hard water is as satisfactory for human consumption as soft waters, their action with soap, their use for cleansing purpose is quite unsatisfactory, unless soap costs are disregarded. Soap consumption by hard waters represents an economic loss to the water user. Sodium soaps react with multivalent metallic cations to form a precipitate, thereby losing their surfactant properties. In recent years these problems have been largely alleviated by the developments of soaps and detergents that do not react with hardness. Boiler scale, the result of the carbonate hardness precipitation, may cause considerable economic loss through fouling of water heater and hot water pipes. Change in pH in the water distribution systems may also result in deposits of precipitates. Bicarbonates begin to convert to the less soluble carbonates at pH values above 9.0. Magnesium hardness, particularly associated with the sulfate ion has a laxative effect on persons unaccustomed to it. Magnesium concentrations of less than 50 mg/l are desirable in potable waters, although many public water supplies exceed the amount. Calcium hardness presents no public health problem. In fact, hard water is apparently beneficial to the human cardiovascular system

Hardness of water can be reduced by (i) Boiling (ii) Lime soda process (iii) Zeolite or Base Exchange process (iv) Ion-exchange process.

EXPERIMENT – 3: Determination of Dissolved Oxygen

Aim: To determine dissolved oxygen of the given water sample.

Apparatus:

- 300 ml BOD bottles
- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Reagents and Indicators Used:

- Manganous sulphate
- Alkali azide
- Concentrated sulphuric acid
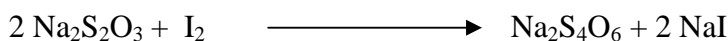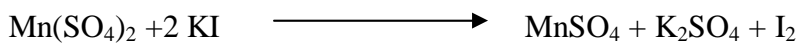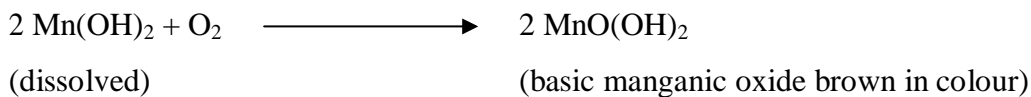- Sodium thiosulphate [0.025N]
- Starch as an indicator

Theory:

Oxygen in various forms is required for the very sustenance of all living organisms and to carry out the all metabolic processes. Oxygen is classed as poorly soluble since it does not react with water chemically and its solubility is directly proportional to their partial pressures. Oxygen solubility also depends on temperature and salinity of water. Hence the solubility of oxygen at a given temperature decreases as one move from fresh water to estuary to the ocean. The solubility of atmospheric oxygen in fresh water ranges from 14.6 mg/l at 0^0C to 7 mg/l at 35^0C at 1 atmospheric pressure.

The dissolved oxygen levels in water decrease due to biological oxidation of organic matter (resulting from discharge of domestic and industrial wastes, agricultural runoff, etc.). Changes in levels of dissolved oxygen in aquatic systems have a detrimental effect on biota in that system. Also, decreased amounts of dissolved oxygen are an indicator of pollution of that particular water body. High concentrations of dissolved oxygen levels in waters used in industries lead to corrosion of pipes. The analysis of dissolved oxygen plays an important role in water pollution control and wastewater treatment processes. Thus, estimation of dissolved oxygen concentration in water and wastewater becomes essential.

Modified Winkler's method or the iodometric method (oxidation-reduction titration) is used for the determination of dissolved oxygen levels.

Principle:

Manganous sulphate reacts with an alkali (KOH or NaOH) to form a white precipitate of manganous hydroxide, which in presence of oxygen gets oxidized to a brown coloured compound (basic manganic oxide). In acidic conditions (on addition of concentrated sulphuric acid), manganese ions are reduced by iodide ions liberates iodine equivalent to the amount of dissolved oxygen present in the sample. The liberated iodine is titrated against sodium thiosulphate solution using starch as an indicator.

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

$$2\ Mn(OH)_2 + O_2 \longrightarrow 2\ MnO(OH)_2$$
(dissolved)   (basic manganic oxide brown in colour)

$$MnO(OH)_2 + 2\ H_2SO_4 \longrightarrow Mn(SO_4)_2 + 3\ H_2O$$
                                                                  (Manganic sulphate)

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

$$2\ Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2\ NaI$$
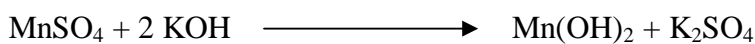
Procedure:

1. Take the BOD bottle and measure the capacity of BOD bottle.
2. Add given sample in a BOD bottle and add 2 ml manganous sulphate and 2 ml alkali azide solution to the BOD bottle.
3. Exclude air by bubbles and mix by repeatedly inverting the bottles about two to three times.
4. If no oxygen is present, the manganous ion reacts with hydroxide ions to form white precipitate of mangonous hydroxide.

$$Mn + 2\ (OH^-) \longrightarrow Mn(OH)_2\ (white\ ppt)$$

If oxygen is present some Mn^{++} ions is oxidized to Mn^{+++} of a precipitate of manganic oxide.

$$Mn^{+++} + 2\ (OH^-) + \tfrac{1}{2}\ O_2 \longrightarrow MnO_2 + H_2O$$

<div align="center">(Brown ppt)</div>

5. After shaking and allowing sufficient time for all oxygen to react, the chemical precipitates are allowed to settle for about 5 minutes.
6. Add 2 ml of concentrated sulphuric acid to the BOD bottle.
7. The contents in the bottle are mixed by inverting until the suspension is completely dissolved and the yellow color develops.
8. Take 203 ml of solution from BOD bottle, this in actual corresponds to 200 ml of the original sample in a conical flask.
9. Titrate it with 0.025 N sodium thiosulphate (Na$_2$S$_2$O$_3$) until yellow color turns to strong pale color.
10. Add 1 to 2 ml of starch solution, blue color develops. Continue the titration to the first disappearance of the blue color.
11. Note down the final burette reading.

Calculations:

To determine,

$$\text{ml of sample to be taken} = \frac{\text{Titration Volume} \times \text{Capacity of bottle}}{\text{Capacity of bottle} - \text{Reagents added}}$$

$$= \frac{200\ \text{ml} \times 300\ \text{ml}}{300\ \text{ml} - 4\text{ml}}$$

$$= 203\ \text{ml}$$

Dissolved Oxygen in mg/l $= \dfrac{\mathbf{V \times Normality \times 8 \times 1000}}{\mathbf{ml\ of\ sample}}$

Where, V = Volume of sodium thiosulphate consumed

      N = Normality of sodium thiosulpahte

      8 = Equivalent weight of oxygen

Tabular Column:

Sl. No	Sample taken	Burette Reading			Average volume of $Na_2S_2O_3$
		Initial reading (IR)	Final Reading (FR)	Volume of $Na_2S_2O_3$ consumed	
1					
2					
3					

Result: Express the results as mg/l dissolved oxygen

Environmental Significance:

It is necessary to know the D.O level to assess quality of raw water and to keep a check on stream pollution. A minimum D.O of 4 to 5 mg/L is desirable for the survival of aquatic life; higher values of D.O may cause corrosion of Iron and steel. Drinking water should be rich in D.O concentration for good taste. D.O test is used to evaluate the pollution strength of domestic and industrial wastes.

Fish and other aquatic life depend on dissolved oxygen present in water for their survival. The aerobic organisms with the help of dissolved oxygen do biological oxidation of organic matter in natural waters and wastewater. Critical conditions occur during summer when solubility of Oxygen is minimum and rate of biological activity is maximum.

In streams and other bodies of water, dissolved oxygen supports fish and other aquatic life. A minimum of 4 mg/L DO must be present in streams for aquatic life. When a stream gets polluted due to discharge of organic wastes, the DO level drops up to zero. This is known as Oxygen sag. Slowly the stream recovers in DO due to self-purification and regains saturation concentration of DO after a long time. DO test is used for stream pollution monitoring. Absence of DO indicates anaerobic conditions. DO determinations serve as basis for BOD determination. All aerobic wastewater treatment processes depend upon the presence of DO.

EXPERIMENT – 4: Determination of Biochemical Oxygen Demand (BOD)

Aim: To determine BOD of the given water sample.

Apparatus:

- 300 ml BOD bottles
- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Reagents and Indicators Used:

- Manganous sulphate
- Alkali azide
- Concentrated sulphuric acid
- Sodium thiosulphate [0.025N]
- Starch as an indicator

Theory:

Biochemical oxygen demand (BOD) is the amount of oxygen required by the microorganisms while stabilizing decomposable organic matter under aerobic conditions. The BOD test is used to determine the amount of oxygen required by of domestic and industrial wastes if discharged into natural water bodies in which aerobic conditions exist. This in turn will give the degree of pollution in lakes and streams at any instant of time and their self purification capacity. BOD test is basically an bioassay procedure since it involves the measurement of oxygen consumed by living organisms (mainly bacteria) while stabilizing the organic matter present in a waste, under conditions as similar as possible to those that occur in nature. The test is carried for 5 days incubation at 20^0C as a major portion of organic matter (70 -80%) is oxidized during this period. The organic matter has both carbonaceous and nitrogenous compounds. The carbonaceous matter is oxidized within the 5 days; hence it is only the carbonaceous matter that is estimated in this test. However, nitrification does not occur to a detectable level during the 5-day period, hence nitrogenous matters are not determined. If nitrification occurs within the stipulated period of 5 days, then BOD estimation should be carried out after suppressing

nitrification with 1 ml of 0.05% thiourea solution per litre of standard dilution water. In tropical countries like India due to the temperature being high, BOD test is to be carried out for 3 days at 37^0C as recommended b y Central Pollution Control Board instead of 5 days at 20^0C.

$$\text{Organic matter} + O_2 \longrightarrow CO_2 + \text{bacteria's} + H_2O + \text{Heat}$$
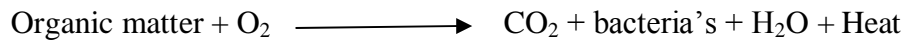
The procedure for sample collection for BOD estimation is same as that for dissolved oxygen. The test should be conducted at the earliest from the time of sample collection and sample should be transported to laboratory at 4^0C. Temperature should be maintained constant during the period of incubation of sample. An increase or decrease of BOD by 4.7% occurs if a slight increase or decrease of even 1^0C takes place.

Principle:

Estimation of dissolved oxygen is the core for BOD determination. Dissolved oxygen content of the sample is determined before and after incubation of 5 days at 20^0C and is calculated from the difference between initial and final dissolved oxygen levels. Samples having less or no dissolved oxygen are to be diluted numerous times with dilution water saturated with oxygen such that sufficient amount of oxygen is available for oxidation to take place.

Method of BOD determination:
Dilution method
Principle:

In this method the waste water sample is diluted suitably with nutrient added and oxygen saturated distilled water. The DO is determined before and after incubation at 20°C for 5 days. The BOD of sample is calculated by knowing the percent dilution.

Procedure:

1. Preparation of dilution water:

Add 1 ml each of Phosphate buffer, $MgSO_4$, $CaCl_2$ and $FeCl_3$ solutions for each liter of distilled water and saturate with dissolved oxygen by aerating.

Phosphate buffer produces and maintains a pH of 7.0. Ca, Mg and Iron salts supply the requirement of mineral and sulphur.

Seeding: When testing industrial waste waters which lack necessary microorganismsabout 2 ml of domestic sewage for each litre of dilution water is added. This supplies the required microorganisms.

2. Preparation of dilution:

BOD is not influenced by DO concentrations as low as 0.5mg/L. DO depletion of less than 2mg/L are not statistically reliable. Therefore the dilutions are so chosen that DO depletion is more than 2mg/L but final DO after incubation is more than 0.5mg/L. This restriction usually means a range of concentration of BOD of 2 to 7 mg/L.

Knowing the approximate value of BOD different dilutions are prepared. The following concentrations are suggested:

Strong Industrial Wastewater: 0.1 to 1 percent

Raw and settled Wastewater: 0.1 to 1 percent

Partially treated Wastewater: 5 to 10 percent

Polluted Wastewater: 25 to 100percent

Ex: Approximate BOD= 200mg/L

Use % dilutions between l% and 3.5%

Take dilution water in a measuring cylinder of 1000-2000ml capacity, filling the cylinder half full. Add required quantity of sample and dilute to appropriate level with dilution water. Mix with a mixing rod without entrainment of air. Fill up two BOD bottles with mixed dilution, one for 5 days incubation and the other for initial DO determination. Direct dilutions also can be made.

Determine the initial DO of the dilution for one bottle. Incubate the other dilution bottle for 5 days at 20°C maintaining a water seal throughout the incubation period. Determine DO of the diluted sample after 5 days incubation. Determine the initial and final DO for other dilutions also.

Blank test: For the dilution water, without the sample, determine initial DO and DO after 5 days incubation. The DO results for the blank serve as a rough check on the quality of dilution water. The DO depletion for the blank should not be more than 0.2mg/L over the incubation period.

Calculation:

$$\text{ml of sample to be taken for titration} = \frac{\text{Titration Volume} \times \text{Capacity of bottle}}{\text{Capacity of bottle} - \text{Reagents added}}$$

$$= \rule{2cm}{0.4pt}\ \text{ml}$$

Tabular Column:

Sample	Bottle No.	Exact Capacity of bottle	Volume of sample taken for titration	Titration Reading			DO in mg/l
				IR	FR	Volume of $Na_2S_2O_3$ consumed	
0th day Sample taken							
0th day Blank							
5th day Sample taken							
5th day Blank							

Dissolved Oxygen in mg/l = $\dfrac{V \times Normality \times 8 \times 1000}{ml\ of\ sample}$

Where, V = Volume of sodium thiosulphate consumed
   N = Normality of sodium thiosulpahte
   8 = Equivalent weight of oxygen

$BOD_5 = [(D_0 - D_5) - (C_0 - C_5)] \times$ Dilution factor BOD_5 =----------- mg/l
   Where,

D_0 – DO in the sample bottle on 0th day

D_5 – DO in the sample bottle on 5th day

C_0 - DO in the blank bottle on 0th day

C_5 – DO in the blank bottle on 5th day

$(D_0 - D_5)$ = (DO depleted in sample + Dilution water)

$(C_0 - C_5)$= DO depression in dilution water

Dilution Factor = $\dfrac{Volume\ of\ diluted\ sample}{Volume\ of\ Undiluted\ sample}$

RESULT: The 5 day BOD for a given sample at 20^0C = ----------------- mg/l

Environmental Significance:

BOD value indicates the strength & Pollution load of waste water. BOD value helps in determining the degree of treatment required, in the design of various treatment units and in deciding the method of disposal. The performance efficiencies of individual treatment units and that of the treatment plant as a whole can also be determined by conducting BOD test on influent and effluent of the treatment unit. Routine operation and maintenance of treatment system is also controlled using BOD test. It is an important test in Pollution control and in stream pollution monitoring. BOD or oxidizable organic matter should be absent in drinking water.

Standard unit of measurement:

BOD is expressed as mg/L. Incubation temperature and time should be mentioned to have a correct idea of BOD value.

Values of BOD for domestic waste

Weak sewage   -up to 200 mg/L

Average sewage   - 200 to 350mg/L

Strong sewage   -350 to 500 mg/L

Permissible limit:

I.   For disposal into a body of water

     For domestic sewage> 30 mg/L

II. For disposal on land,

     Not more than 500 mg/L (Refer IS codes)

EXPERIMENT – 5: Determination Chlorides

Aim: To determine chlorides present in a given water sample.

Apparatus:

- 250 ml conical flasks
- 100 ml, 1000 ml volumetric flasks
- Pipettes
- Burette

Reagents and Indicators Used:

- N/35.5 or 0.02 N Silver Nitrate solution, Potassium Chromate as indicator

Theory:

Chloride is the major anion commonly found in all natural waters and its concentration widely ranges from a few milligrams to thousands of milligrams. Chloride concentration increases proportionately with mineral content. Fresh waters have low concentration of chlorides in comparison to marine waters. High concentrations of chlorides in waters are due to the anthropogenic activities such as discharge of sewage and industrial effluents; intrusion of sea water; leachates from landfills etc,. Higher amounts of chlorides cause a change in taste of drinking water, corrosion and palatability. Chlorides as such are not harmful to human beings but the cations like calcium, sodium, magnesium or potassium associated with it may affect the health of an individual who has a medical history of heart or kidney ailments. Presence of high concentrations of chlorides in waters can have harmful effect on metallic pipes leading to corrosion and cause toxic effects on plants.

Argentometric (Silver nitrate) method

Principle:

Under neutral to slightly alkaline conditions, silver nitrate reacts with chlorides and precipitates as silver chlorides. The completion of the reaction is indicated by the reaction of silver nitrate with potassium chromate solution as indicator.

$$AgNO_3 + Cl^- \longrightarrow AgCl \downarrow + NO_3$$
$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2 KNO_3$$
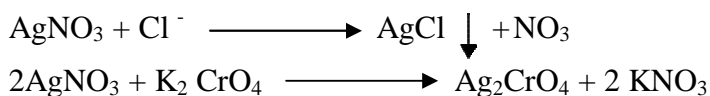
Procedure:

1. Take 100 ml sample in a 250 ml conical flask.

2. Check the pH of the sample. If the pH is within 7-9, the sample can be directly titrated. If the pH is not within the range, adjust the pH using 1N H_2SO_4 or 1N NaOH.

3. Add 1 ml of potassium chromate indicator solution to the sample.

4. Mix well the contents in the flask by swirling.

5. Sample turns to yellow color, and titrates with 0.02 N $AgNO_3$ solution slowly with constant stirring till the end point is reached as brick red color.

6. Note down the final burette reading.

7. Conduct a blank test by using 100 ml chloride free distilled water. Note down the initial and final burette readings.

Calculations:

Tabular Column: (For sample and blank)

SL NO.	Sample taken	IR	FR	Volume of 0.02 N $AgNO_3$ consumed	Average Volume of 0.02 N $AgNO_3$ consumed
1					
2					
3					
4					

$$\text{Chloride concentration} = \frac{(A - B) \times \text{Normality} \times 35.45 \times 1000}{\text{ml of sample taken}}$$

Where,

A = Volume of titrant consumed sample test

B = volume of titrant consumed in blank test

Result: The concentration of chloride in a given sample =------------ mg/l as Cl⁻.

Significance:

Chloride, associated with sodium exert salty taste, when its concentration is more than 250mg/L. for this reason, chlorides are generally limited to 250mg/L in supplies intended for public use. Excess chlorides can also corrode concrete by extracting calcium in the form of calcite.

EXPERIMENT – 6: Determination of percentage of available chlorine in Bleaching powder and Determination of Residual Chlorine

a) Determination of Percentage of available chlorine in bleaching powder

Aim: To determine the percentage of available chlorine in bleaching powder

Apparatus:

- □ 250 ml conical flasks
- □ 100 ml, 1000 ml volumetric flasks
- □ Pipettes
- □ Burette

Reagents and Indicators Used:

- □ Glacial acetic acid
- □ Potassium iodide (powder)
- □ 0.1N Sodium thiosulphate
- □ Starch as an indicator
- □ Bleaching powder, Potassium Iodide (Powder), 0.025N Sodium Thiosuiphate reagent, Glacial acetic acid, Starch indicator

Theory:

Bleaching powder is hydroscopic and contains calcium, oxygen and chlorine.

$$CaOCl_2 + H_2O \longrightarrow Ca(OH)_2 + Cl_2$$
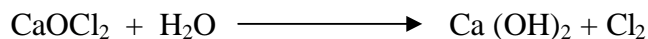
Bleaching powder losses its chlorine content, when it is exposed to the atmosphere due to prolonged use and the instability of chlorine molecules. This test is useful to measure the capacity of bleaching powder. It is also useful to estimate the exact amount of bleaching powder required for effective disinfection of water.

Bleaching powder is used as a disinfectant in small scale water treatment plants. The commercial bleaching powder contains low values of available chlorine (25-30%). Available chlorine further reduces if the powder is exposed to atmosphere or stored for a long period.

To determine the dosage of Bleaching powder its strength should be determined by finding the percentage available chlorine.

Bleaching powder is hypochlorite of Calcium. $Ca(OCl)_2$. It combines with water and liberates hypochlorite ion, which forms hypochlorous acid when it combines with hydrogen ions present in water.

$$Ca(OCl)_2 \square Ca^{++} + 2OCl^-$$

$$Ocl^{++} + H^+ \square HOCl$$
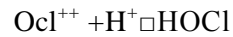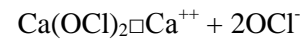
The hypochlorous acid acts as a disinfectant.

Chlorine content in bleaching powder can be determined by iodometric method or by Orthotolidine method.

Procedure:

1. Weigh out accurately 250 mg or 2.5 gm of bleaching powder.

2. Add 10 ml of distilled water with bleaching powder in a china dish and make a paste.

3. Transfer the milky paste into a 250 ml standard flask.

4. Dilute the solution up to the mark in standard 250 ml flask with distilled water and mix well.

5. Transfer 25 ml of bleaching powder solution into a clean conical flask using measuring cylinder.

6. Add 5 ml of glacial acetic acid and 1 gm of potassium iodide and mix well.

7. Keep it for a minute and titrate against 0.1N sodium thiosulphate solution till light yellow color appears.

8. Now add 1 ml of starch solution as indicator and blue color appears.

9. Then titrate against 0.025N $Na_2S_2O_3$ till dark blue color disappears in a solution.

Calculations:

Tabular Column:

SL. NO.	Sample used	Burette Reading			Average Volume of 0.025 N $Na_2S_2O_3$ used
		IR	FR	FR - IR	
1	Bleaching Powder solution				
2	Bleaching Powder solution				
3	Bleaching Powder solution				

Calculations:

Normality of BP × Volume of BP = Normality of $Na_2S_2O_3$ × Volume of $Na_2S_2O_3$ N_{BP} × V_{BP} = N of $Na_2S_2O_3$ × V of $Na_2S_2O_3$

$$N_{BP} = \frac{\text{N of } Na_2S_2O_3 \times \text{V of } Na_2S_2O_3}{V_{BP}}$$

$$N_{BP} = \text{---------------------}$$

Weight of Chlorine in BP/250ml = $\dfrac{N_{BP} \times 35.45}{4}$

$$= \text{----------------gm}$$

Percentage of chlorine in BP = W$\dfrac{\text{eight of chlorine in BP} \times 100}{\text{Weight of BP taken}}$

$$= \text{--------------------}\%$$

Result: Percentage of available chlorine in Bleaching Powder = ----------------- %

Environmental Significance:

Chlorine is available in different states, gaseous, liquid and also as a solid. Bleaching powder is a slaked lime through which chlorine is introduced into water. This test is useful to assess the quality of bleaching powder. Also it is useful to estimate the amount of bleaching powder required for effective disinfection of water.

b) Determination of Residual Chlorine

Aim: To determine the residual chlorine in given sample

Apparatus:
- □ 250 ml conical flasks
- □ 100 ml, 1000 ml volumetric flasks
- □ Pipettes
- □ Burette

Reagents and Indicators Used:
- □ Glacial acetic acid
- □ Potassium iodide (powder)
- □ 0.025Sodium thiosulphate
- □ Starch as an indicator
- □ Bleaching powder, Potassium Iodide (Powder), 0.025N Sodium Thiosuiphate reagent, Glacial acetic acid, Starch indicator
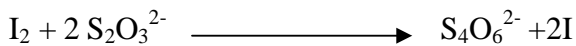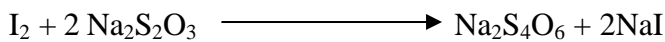
Theory:

Disinfection of public water supplies and treated wastewaters by chlorine is a common employed to prevent the spread of water borne diseases such as cholera, typhoid,

gastroenteritis and free chlorine. Chlorine in both the forms as a strong oxidizing agent and

frequently dissipates rapidly that very less disinfection takes place until large amounts of chlorine demand have been added.

Chlorine may be present in water as free available chlorine (as hypochlorous acid or hypochlorite ions or both) and /or as combined available chlorine, hypochorous acid and hypochlorite ions are commonly referred to as free chlorine residuals and the chloramines are called combined chlorine residuals. At lower pH, free chlorine residuals favor the formation of HOCl instead OCl⁻, which is more effective as a disinfectant.

Principle: This method depends on the oxidizing power of free and combined chlorine residuals to convert iodide ion to free iodine. Free and available chlorine residuals liberates free iodine on reacting with potassium iodide and the released iodine is titrated with standard sodium thiosulphate solution (reducing agent) using starch as an indicator and the end point is indicated with the disappearance of blue colour.

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

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

$$I_2 + 2\,S_2O_3^{2-} \longrightarrow S_4O_6^{2-} + 2I$$

The dose of chlorine which yields residual chlorine of about 0.2mg/L at the end of 10 minutes contact period is considered as the optimum dosage.

Procedure:

1. Take 100 ml of sample in a 250 ml conical flask.
2. Add 1gm of potassium iodide crystals.
3. Add 10 ml of glacial acetic acid; mix well with a glass rod.
4. Titrate the sample with standard N/40 or 0.025 N sodium thiosulphate solution until the yellow color of liberated iodide is almost faded out.
5. Add 2ml of starch indicator, continue the titration with N/40 solution of sodium thiosulphate, till dark blue color disappears. Note down the volume of titrant used.
6. Repeat the same procedure for distilled water as blank test and note down the titrant volume.

Calculation:

Tabular Column: (for sample and blank)

SL. NO.	Sample taken	Burette Reading			Volume of Na$_2$S$_2$O$_3$ used
		IR	FR	FR - IR	
1					
2					
3					
4					

Residual Chlorine in mg/l = $\dfrac{\text{V} \times \text{Normality} \times 1000 \times 35.45}{\text{ml of sample taken}}$

= .... **mg/l**

Result:

EXPERIMENT – 7: Determination Solids in Sewage

Aim: To determine the total solids, suspended solids, dissolved solids, volatile solids, fixed solids and settleable solids in sewage.

Apparatus:

- Porcelain dish,
- Crucibles,
- Imhoff cone,
- Drying oven,
- Analytical balance,
- muffle furnace,
- Desiccator

Theory:

Solids can be defined as the substance that remains as the residue on evaporation and drying at a definite temperature. Solids are present in both suspended and dissolved form in water. The amount of suspended solids will be more in domestic sewage and industrial wastewater whereas dissolved solids accounts to be high in potable water. Presence of high dissolved solids renders the water unfit for drinking, agricultural and industrial purposes.

The significance of determination of suspended solids will be more in case of wastewater from domestic and industrial sectors. While the determination of total dissolved solids is significant in the analysis of potable water.

Solids are estimated as total solids, dissolved solids, suspended solids and settleable solids. In addition to these, fixed and volatile forms are determined for total, dissolved and suspended solids.

a) Total Solids

Total solids are those matters that are in suspension and in dissolved form.

Principle: A known quantity of well mixed sample is evaporated to dryness in a dish.

The difference in the weight of the dish is the amount of total solids in the sample.

Apparatus:

- Platinum dish (for water), Porcelain (sewage/industrial effluents)
- Water bath
- Analytical balance

Procedure:

1. Clean the evaporating dish, dry in an oven at the temperature that is required for the residue; cool in a desiccator and weigh the dish (W_1).

2. Take 50 ml of sample in pre-weighed dish.

3. Evaporate to dryness on a water bath. Further dry the dish with the residue at 105^0C in an oven for one hour.

4. Cool the dish in a desicator.

5. Note the weight of the dish along with the residue (W_2).

Calculations:

Total solids (mg/l) = $\dfrac{(W_2 - W_1) \times 10^6}{V}$

Where, W_1 = initial weight of the dish

W_2 = weight of the dish after evaporation to dryness (mg)

V = Volume of the sample taken (ml)

Result: The results are expressed in mg/l.

b) Dissolved Solids and Suspended solids
Procedure:

1. Determine the initial weight of a clean dry empty crucible (W_3 gms).

2. Take 100 ml of sample in a weighed dry crucible.

3. Filter a known sewage sample through the filter paper number 41 to 44.

4. Keep the crucible in the oven at 103^0C to 105^0C for an hour to remove the water content and dry the solid residue.

5. Cool to room temperature in desiccators and find the final weight of crucible (W_4 gms).

Calculations:

Dissolved solids, mg/l = $\dfrac{(W_4 - W_3) \times 10^6}{\text{ml of sample}}$

= ....mg/l

Department of Civil Engineering, A.I.T, Chikkamagaluru

Suspended Solids = Total Solids – Dissolved Solids

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

c) Fixed and Volatile Solids

Determine the weight of the dish with solid residue (W2 grams) after cooling to room temperature in desiccators (already determined in total solids). Keep the dish in the muffle furnace at 550°C for 15 minutes. Cool to room temperature in desiccators and determine the final weight of dish with fixed residue (W5 grams).

$$\text{Fixed solids} = \frac{(W_5 - W_1) \times 10^6}{\text{ml of sample}}$$

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

Volatile Solids, mg/L = Total solids - Fixed solids

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

d) Settleable solids:

Fill an Imhoff cone up to 1000ml mark with a sewage sample and keep in a stand. Allow settling 1 hour. Gently rotate the cone to dislodge solids sticking to sides of cone. Allow the contents to settle. At the end of settling note the volume of sludge settled at the bottom of cone in ml. Record the volume of settleable matter in the cone as ml/litre.

Results and discussion:

Sl. No	Item	Result
1	Total Solids, mg/l	
2	Suspended Solids, mg/l	
3	Dissolved Solids, mg/l	
4	Fixed solids, mg/l	
5	Volatile solids, mg/l	
6	Settable Solids, ml/l	

EXPERIMENT – 8:

Determination of Turbidity by Nephelometer

Aim: To determine the turbidity of the given water sample.

Apparatus: Jackson candle turbidirmeter, Nephelometer, beaker, pipette.

Reagents: Turbidity free water. Stock turbidity suspension, Standard turbidity suspension.

Theory:

Turbidity in water is caused by the presence of suspended matter, such as clay; silt, finely divided organic and inorganic matter, Plankton and microscopic organisms.

Turbidity of water is an optical property, which causes light to be scattered and absorbed rather than transmitted in straight lines through the sample. Turbidity of water varies during different seasons. It is more during rainy season.
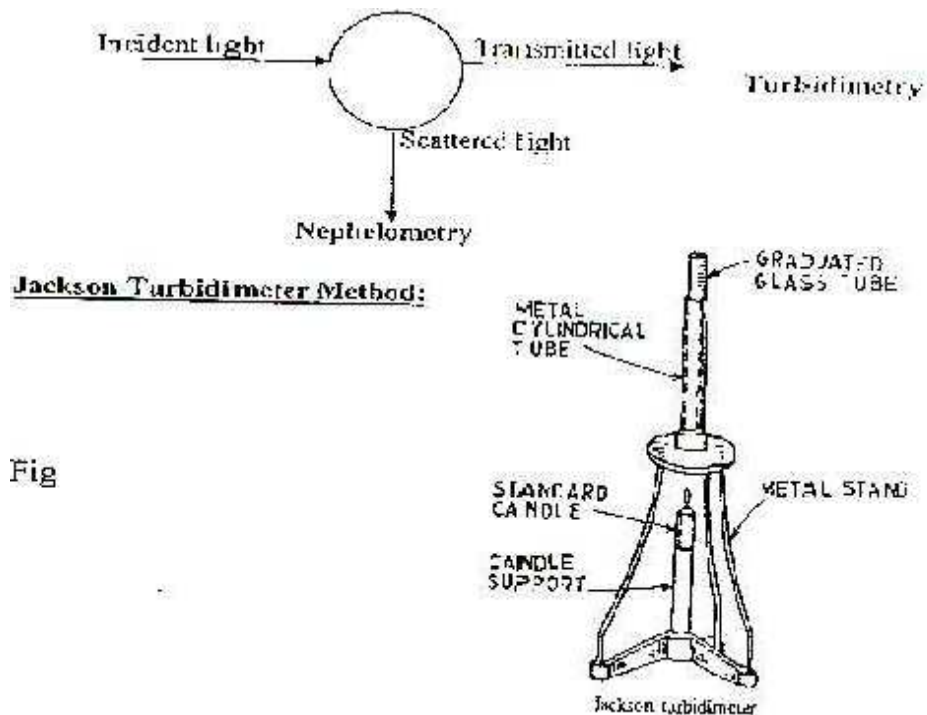
Methods of determination:

i) Visual methods:

    a. Jackson turbidimeter Turbidity range- 100- 1000 (Short tube) 25 -1000 (long tube)

    b. Bottle standards. 5-100 mg/L

ii) Instrumental methods: Turbidimetry and Nephelometry - For measuring low values of turbidity - 0 to 40 units.

A) Jackson turbidimeter

Fig

Principle:

The interference to light path by the turbidity causes the image of a candle to disappear. The image becomes in distinguishable against the background il1umnaton.

Apparatus:

The instrument consists of a calibrated glass tube (short or long), a standard candle and a tripod stand. The candle support consists of a spring-loaded cylinder to keep the top of candle pressed against the top of support as candle bums. The top of support for candle must be 7.6 cms below the bottom of glass tube,

The candle is made of beeswax and designed to burn within the limits of 114 to 126 grains per hour.

Procedure:

Pour the well-mixed sample into the glass tube until the image of the candle flame just disappears from view. The image becomes indistinguishable or merges with the surrounding field. Now remove about 1 percent of sample to make the flame image visible again. Using a pipette slowly add the sample till the image just disappears. Take out the calibrated glass tube and read the turbidity of water sample corresponding to water level in the tube.

Precautions:

1. The glass tube bottom must be kept clean and free from scratches.
2. To keep the candle flame at a constant distance from glass tube frequent trimming of candlewick is necessary.
3. Air draft must be eliminated during measurement to prevent flickering of candle flame.
4. The candle must bum for not more than a few minutes at a time as the flame has a tendency to increase in size.

B) Bottle Standards Method:

Turbidity measurements in the range of 5 to 100 JTU are usually made by reference to Standard suspensions. Standard suspensions must be prepared using Jackson turbidimeter. The standard suspensions of lower turbidity are prepared by dilution with distilled water. The volume of stock required to prepare a particular standard suspension is obtained by using the flowing formula.

$$\text{ml of stock required} = \frac{\text{required std turbidity}}{\text{Stock turbidity}} \times \text{ml of std required}$$

Example: Given Stock turbidity 200 mg/L

                 Required Std turbidity 5 mg/L

                     Std volume = 500 ml

$$\text{ml stock required} = \frac{5 \times 500 \text{ ml}}{200} \quad \Box \quad 12.5 \text{ ml}$$

Procedure:

Compare the shaken sample with standard suspensions. Place the sample and the standards in bottles of the same shape size and type. Leave enough empty space at the top of bottle to allow adequate shaking before each reading. Compare the sample and standards by looking through the sides of the bottles, by looking through them at the same object. Record the turbidity of the sample as that, of the standard, which produces the same, or nearest visual effect.

C) Nephelometer Method:

Low values of turbidity are measured accurately by Nephelometric method. This method uses the principle of Nephelometry. Turbidity of the sample depends on the amount of scattering of light through the sample tube.

Procedure:

Switch on the instrument. Select the appropriate range of turbidity, say 0-100 NTU. Take distilled water in the tube and set zero. Take standard suspension of 100 NTU and standardize. Take sample in the tube and read the turbidity value. Stronger samples are analyzed by taking a suitable dilution.

Results and discussion:

Environmental Significance:

1. Aesthetic: Turbidity in drinking water is usually associated with sewage contamination, by laymen consumers and discourages the use of that water.

2. Filterability: Filtration of water is more difficult and costly when turbidity is more. High turbidity shortens filter run and increases cleaning cost.

3. Disinfection: Turbid particles protect pathogenic organisms from the act ion of disinfecting agents such as chlorine or ozone. Contact between organisms and disinfectant is reduced.

Standard unit of turbidity:

One standard unit of turbidity is that produced by 1 mg of finely divided silica in 1 liter of water.

Permissible limit: 5 to 10 mg/L on silica scale. Excess turbidity from water is removed by plain sedimentation, coagulant aided sedimentation and by filtration.

EXPERIMENT – 9:
Determination of Optimum Dosage of Alum using Jar test apparatus.

Aim: To determine the optimum dosage of Alum for the given water sample by conducting Jar test.

Apparatus: Jar test apparatus, Jackson turbidimeter, Nephelometer, beakers, pipette

Reagents: One percent alum solution.

Theory:

Excess turbidity of water is removed by plain sedimentation or more efficiently by coagulant aided sedimentation. A coagulant like alum dissolves in water and reacts with natural or added alkalinity and releases positively charged hydroxide ions, which attract and neutralize the negatively charged colloidal particles present in water. The colloidal particles agglomerate and form bigger and heavier floe particles, which easily settle down. Coagulation of water takes place in two stages:

1. **Flash mixing** or rapid stirring for 2 to 4 minutes, when the coagulant comes intimately in contact with water and neutralizes the negative charge of colloidal particles.

$$Al_2(SO_4)_3, 18H_2O + Alkalinity \square Al(OH)_3$$

2. **Flocculation**- Slow stirring for about 20 to 30 minutes causes and growth of floe particles which settle down easily when quiescent (still) conditions are provided.

Optimum dosage of a coagulant is the least or economical dosage which bring about effective coagulation and reduction of turbidity of a water sample.

Jar test is a simple method of determining the optimum dosage of a coagulant for a given water sample. It can be also used to study the various parameters affecting coagulation, such as temperature, pH, alkalinity, mixing, different coagulants and coagulant aids.

Jar test apparatus consists of a set of 4 or 6 stirrers with paddles, which can be rotated simultaneously by connecting to a motor and regulator. The speed of paddles can be varied using the regulator. Equal volumes of water are taken in beakers, to which varying doses of coagulants are added. The mixtures are rapidly stirred initially for about 1 minute and slowly stirred for 9 minutes. After 10 minutes of settling, supernatant is decanted from all the jars to measure turbidity. The dose, which gives the least turbidity, is taken as the optimum dosage.
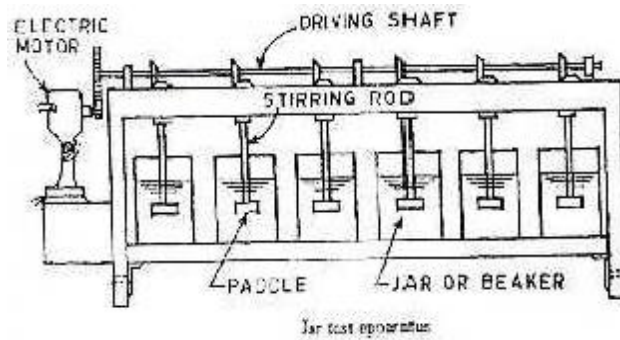
Jar test apparatus

Procedure:

Measure the initial turbidity of the water sample using Jackson turbidimeter or Nephelometer. Measure and adjust the pH to 8 or above. Transfer 500 ml of sample to each beaker. Switch on the Jar test apparatus and adjust the speed of paddles to 100 rpm Add varying doses of alum solution in increasing order to the beakers simultaneously. Allow rapid stirring for one minute. Decrease the speed to 40 rpm and allow slow stirring for 9 minutes. Switch off the apparatus and allow 10 minutes of settling. Take supernatant samples from each beaker and measure turbidity. Plot a graph of settled water Vs Alum dose. Note down optimum dose of alum corresponding to the trough of graph.
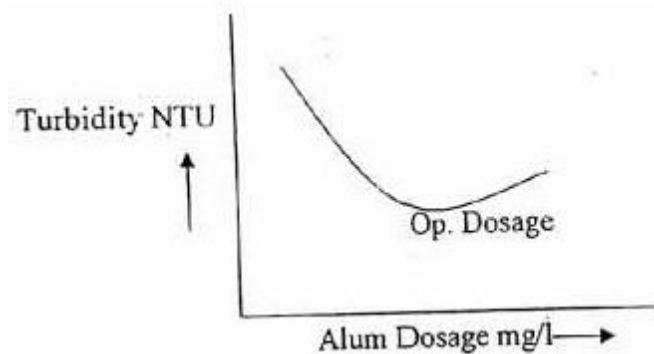
Results and discussion:

EXPERIMENT – 10:
Determination of sodium and potassium using flame photometer.

Aim: To determine the sodium and potassium using flame photometer

Apparatus:

- Flame photometer
- Borosil glassware's

THEORY:

Sodium (Na): Sodium makes up about 2.6% by weight of the earth's crust, making it the 6th most abundant element overall and the most abundant alkali metal. For billions of years sodium is washed out from rocks and soils, ending up in oceans, where it may remain for about 50×106 years. Seawater contains approximately 11,000 ppm of sodium. Rivers contain only about 09 ppm or mg/L of sodium. Drinking water usually contains about 50 ppm or mg/L of sodium. This value is clearly higher for mineral water. In soluble form sodium always occurs as Na ions. Elementary sodium reacts strongly with water, according to the following reaction mechanism:

$2Na(s) + 2H_2O \rightarrow 2NaOH(aq) + H_2(g)$

A colourless solution is formed, consisting of strongly alkaline sodium hydroxide (caustic soda) and hydrogen gas. This is an exothermic reaction. A number of of water solubility of sodium are available. The most familiar sodium compound is sodium chloride (NaCI), otherwise known as kitchen salt or common salt. For NaCI at 20°C the solubility is 359 gmIL, in other words adequately water soluble. Solubility is nearly temperature independent. Sodium is present in the human body in amounts of about 100 grams. It is a dietary partially responsible for nerve functions. Blood serum contains 3.3 gm/L sodium. It regulates extra cellular fluids, acid-base balance and membrane partially together with potassium. One may overdose on sodium from salt. This causes increased blood pressure, arteriosclerosis, oedema, confusion and increased risk of infection from excessive Na intake. Sodium shortages may lead to dehydration, convulsion, muscle paralysis, decreased growth and general numbness. Generally, humans require about 300 mg/day sodium chloride to warrant a balanced sodium level. People that have diarrhoea or other health effects that increase salt requirements need a higher dietary amount of sodium than usual.

People with heart and kidney disease are recommended a sodium poor diet. The high sodium content is the deterioration of the physical condition of the soil (formation of crusts, water logging, and reduced soil permeability. The water infiltration problem occurs within the top few centimeters of the soil and is mainly related to the structural stability of the surface soil. To predict the infiltration problem, the Sodium Absorption Ratio (SAR) is often used. To remove sodium chloride from water, one may apply reverse osmosis, electro dialysis, distillation techniques or ion exchange. Reverse osmosis is most economical considering energy and money requirements.

Potassium (K): Elemental potassium is does not occur in nature because it reacts violently with water. As various compounds, potassium makes up about 1.5% of the weight of the earth's crust and is the 7th most abundant element. Potassium may be detected by taste because it triggers 3 of the 5 types of taste sensations, according to concentration. Dilute solutions of potassium ion taste sweet (allowing moderate concentrations in milk and juices), while higher concentrations become increasingly bitter/alkaline, and finally also salty to the taste. Potassium ions are an essential component of plant nutrition and are found in most soil types. Its primary use in agriculture, horticulture and hydroponic culture is as a fertilizer as the chloride (KCI), sulfate (K_2SO_4) or nitrate (KNO_3). Seawater contains about 400 ppm of potassium. It tends to settle, and consequently ends up in sediment mostly. Rivers generally contains about 2 to 3 ppm potassium. This difference is mainly caused by a large potassium concentration in oceanic basalts. It reacts with water more rapidly than does sodium, which is placed higher in the periodic chart. Potassium is a dietary requirement for humans, and they take up about 1-6 gm/day at a requirement of 2-gm/day. The total potassium amount in the human body lies somewhere between 110 and 140 grams and mainly depends upon muscle mass. Potassium may be removed from water by means of reverse osmosis.

a)   **Aim**: Estimation of Sodium

 Apparatus: Flame Photometer with flame accessaories, Glasswares

   Principal: the sample solution is sucked by an atomiser under controlled conditions. The radiation from the flame enters a dispersing device in order to isolate the desired region of the spectrum. The intensty of isolated radiation can be measured by a phototube. After carefully cslibrating the photo meter with solution of known composition and concentration, it is possible to

correlate the intensity of a given spectral line of the unknown with the amount of an element present that emits the particular radiation.

Reagents and Preparation:

1. Deionised distilled water.

2. Stock sodium solution: Dissolve 2.542g NaCl and dilute to 1000N with distilled water 1.00ml = 1.00mg of sodium.

3. Standard sodium solutions: Prepare a series of working standard solutions in the range of 20, 40, 60, 80 and 100mg/L by diluting 2,4,6,8 & 10ml of stock sodium solution to 100ml using distilled water.

Procedure:

1. Select Sodium filter with the help of filter selector of the burner unit of flame photometer. Ignite the burner and adjust the air supply pressure between $0.3 - 0.6$ Kg/cm2 and gas supply so as to get blue cone flame in the burner.

2. Feed distilled water to the atomizer, wait at least for 30 seconds and adjust meter reading to zero.

3. Run the standard solutions first adjusting the meter reading to 1.0 by using 100mg/L standard solution. Feed 20, 40, 60 & 80 mg/L standard solutions, taking care to run distilled water between standard runs and ensuring meter shows zero reading.

4. Note the respective readings for 20, 40, 60, & 80mg/L standard solutions.

5. Run the filtered water samples, diluting if necessary with distilled water and note down both dilution factor and reading.

6. A calibration curve is plotted using the standard readings.

7. Sodium values for samples are calculated using the calibration curve.

8. If '1' is observed on the display screen, dilute the sample with distilled water for Suitable proportion.

b) **Aim**: Estimation of Potassium

Apparatus: Flame Photometer with flame accessaories, Glasswares

Principal: Potassium is determined by direct reading type of flame photometer. The sample is sprayed into a air-fuel mixture flame and excitation is carried out under carefully controlled and reproducible conditions. The intensity of emitted light is measured by a phototube. The intensity of light is proportional to the concentration of the element. The calibration curve may be linear but has a tendency to level off at higher concentration.

Reagents and Preparation:

1. Deionised distilled water.

2. Stock Potassium solution: Dissolve 1.91gm KCl and dilute to 1000ml with distilled water. This solution is 1ml = 1mg of potassium.

Standard Potassium solutions: Prepare a series of working standard solutions in the range of 20, 40, 60, 80 and 100mg/L by diluting 2,4,6,8 & 10ml of stock potassium solution to 100ml using distilled water

Procedure

1. Select potassium filter with the help of filter selector of the burner unit of flame photometer. Ignite the burner and adjust the air supply pressure between 0.3 – 0.6 Kg/cm2 and gas supply so as to get blue cone flame in the burner.

2. Feed distilled water to the atomizer, wait at least for 30 seconds and adjust meter reading to zero.

3. Run the highest concentration standard solutions first adjusting the meter reading to 100 by using 100 mg/L standard solution. Feed 20, 40, 60 & 80 mg/L standard solutions, taking care to run distilled water between standard runs and ensuring meter shows zero reading.

3. Note the respective readings for 20,40,60, & 80mg/L standard solutions.

4. Run the filtered water samples, diluting if necessary with distilled water and note down both dilution factor and reading.

5. A calibration curve is plotted using the standard readings.

6. Potassium values for samples are calculated using the calibration curve.

Calculation

Potassium mg/L = A X D

Where, A = Concentration in ppm from the graph corresponding to instrument reading.

D = Dilution factor if sample is diluted

4. Deionised distilled water.

5. Stock Potassium solution: Dissolve 1.91gm KCl and dilute to 1000ml with distilled water. This solution is 1ml = 1mg of potassium.

6. Standard Potassium solutions: Prepare a series of working standard solutions in the range of 20, 40, 60, 80 and 100mg/L by diluting 2,4,6,8 & 10ml of stock potassium solution to 100ml using distilled water.

Results and Discussion:

Sl. No.	Parameters Analyzed	Results

EXPERIMENT –11: Determination of Nitrates by Spectrophotometer

Aim -To determine the amount of nitrate present in given waste water sample

Apparatus – Spectrophotometer, Cuvettes

**Instruments used-** Spectrophotometer

Reagents required-

□ Nitrover 5 nitrate reagent

Principle-Nitrate present in the sample reacts with PDA to produce a nitro derivate , which in alkaline solution ie after the addition of ammonia develops a yellow colour. The development of yellow colour is attributed to rearrangement in the structure of nitro derivate. The colour produced follows Beer's law and is proportional to the concentration of nitrate present in the sample. The concentration of nitrate is determined using spectrophotometer at the maximum wavelength absorption between 410 nm and 480 nm.

Theory- Total nitrogen is comprised of organic nitrogen, ammonia nitrogen nitrite and nitrate. Organic nitrogen is determined by the kjeldahl method where kjeldahl nitrogen is the total of the organic and ammonia nitrogen. Ammonia nitrogen exist in aqueous solution as either ammonium ion or ammonia depending on the PH of the solution with the following reaction

$NH_3 + H_2O = NH_4 + OH^-$
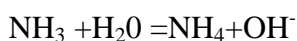
At PH level above 7, the equilibrium is displaced to the left. At PH level below 7 the ammonium ion is predominant. Ammonia is determined by raising the PH by distilling off the ammonia with the steam produced

$N_2O_3 + H_2O = 2NO_3^- + 2H^+$
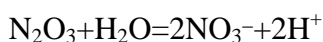
Nitrate nitrogen is unstable and is easily oxidized to the nitrate form it is an indicator of past pollution in the process of stabilization of components and seldom exceeds 1mg/l in waste water or 0.1mg/l in surface water or waste water

$N_2O_5 + H_2O = 2NO_3^- + 2H^+$
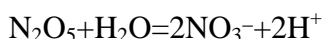
Nitrate nitrogen is the most highly oxidized form of nitrogen found in waste water where secondary effluents are reclaimed for ground water recharge.

Procedure

- ☐ Enter the program number for nitrate i,e 365.

- ☐ Then the display will show the dial nanometer to 500 nm. Rotate the wavelength dial until the display show 500 nm.

- ☐ Take two numbers of 25 ml cuvettes and fill the cuvette with sample.

- ☐ Add the content of nitrover -5 into one of the cuvette. Swirl to dissolve. A white turbid solution will be developed if nitrate is present.

- ☐ Press shift timer for one minute. Shake the sample well until the timer beeps.

- ☐ Now again press shift timer for reaction time of 5 minutes. An amber color will be developed if nitrate nitrogen is present.

- ☐ Place the cuvette which contains blank solution in the cell holder and close the lid. Press zero button, then the display shoes zero (0) reading.

- ☐ Now Place the cuvette contains sample in the cell holder and press read button, then the display shows directly reading of nitrate content concentration in mg/L.

Observation and calculations

Permissible limit of nitrate = 45 mg/L

Sample details	Nitrate Concentration in mg/L

Environmental significance

- ☐ Acts as an indicator of sanitary quality

- ☐ The importance of oxidation in rivers can be found

- ☐ Helps to solve nutritional related problems

- ☐ Aids in controlling biological treatment processes

EXPERIMENT –12: Determination of Iron and Manganese

Aim-To determine the amount of iron present in a given water sample

Apparatus -Spectrophotometer, Cuvettes

Reagents – Ferrover iron

Principle

Phenanthrolin method is the standard procedure for measurement of iron in water, except when phosphate or heavy metal interference is present. The method depends on the fact that 1,10 phenanthrolin solution combines with Fe^{2+} to form a red complex iron. The colour produced confirms to Beer's law and is measured by photometric comparison. The ferric state of iron is oxidized and reduced to ferrous state in the presence of HCl which results in formation of pellets. When these pellets comes in contact with 1,10 phenanthroline PH 3.2-3.3. forms a red complex the intensity is directly proportional to the concentration of iron. This intensity is measured at 510nm using spectrophotometer.

Procedure

- □ Enter the program number for iron i,e 265.
- □ Then the display will show the dial nanometer to 510 nm. Rotate the wavelength dial until the display show 510 nm.
- □ Take two numbers of 10 ml cuvettes and fill the cuvette with sample.
- □ Add the content of Ferrover into one of the cuvette. Swirl to dissolve. Press shift timer for three minute. Shake the sample well until the timer beeps.
- □ Place the cuvette which contains blank solution in the cell holder and close the lid. Press zero button, then the display shoes zero (0) reading.
- □ Now Place the cuvette contains sample in the cell holder and press read button, then the display shows directly reading of iron content concentration in mg/L.

Sample details	Nitrate Concentration in mg/L

Result: The amount of iron present in given sample is _____

Comments

The acceptable limit of iron is 0.1mg/l and maximum permissible limit is 1mg/l

Environmental significance

Human by consuming water containing iron suffer from no harmful effects, when such water is exposed to air become turbid and unacceptable

EXPERIMENT –13: Determination of COD

Aim- To determine the COD of waste water sample

Apparatus- Pipettes, beakers, vials, measuring cylinders, micro burrete, COD digester

Reagents-

- □ Standard potassium dichromate ($K_2Cr_2O_7$) digestion solution 0.1 N
- □ Sulphuric acid reagent
- □ Ferroin indicator
- □ Standard ferrous ammonium sulphate solution (0.1M)

Theory: COD is defined as the quantity of a specified oxidant that reacts with the sample under controlled conditions. The quantity of oxygen consumed is expressed in terms of its oxygen equivalence.COD is a measure of oxygen equivalent of the organic water content of a sample. It is susceptible to oxidation by a strong chemical oxidant. this test allows the measurement of waste in terms of oxygen required for oxidation of organic matter containing co2 and water. It is based on the fact that all organic compounds with a few exceptions may be oxidized by action of strong oxidizing agent under acidic condition. The Dichromate reflux method is preferred over procedures using other oxidants because of its superior oxidizing ability, applicability to a wide range and variety of sample and ease of manipulation. Oxidation of most organic samples is 95-100% of theoretical value

Principle: $K_2Cr_2O_7$ digestion solution is a strong oxidizing agent used to stabilize 0.1M of reagent in sulphuric acid at elevated temperature values. Dichromate solution stabilizes the solution used to determine the molecular oxygen used from oxidant solution. Many organic substances that are difficult to oxidize biologically can be oxidized chemically and it is titrated against standard FAS solution to know the amount of $K_2Cr_2O_7$ left in the presence of ferroin indicator

Procedure

- □ Transfer 2.5 ml of sample to vial
- □ Add 1.5ml of $K_2Cr_2O_7$
- □ Add 3.5ml of H_2SO_4 reagent down inside of vial
- □ Tight the cap of the vial and invert it several times to mix completely.

□ In the same manner reflex and titrate a blank containing the reagent and volume of distilled water equal to that of sample , place the vial in COD digester , preheated to 150 degree Celsius and reflux for 2 hr, cool to room temperature. Titrate against 0.1M FAS by adding 1 or 2 drops of Ferroin indicator.

□ Sharp color changes from straw yellow to reddish brown

Calculation

Observations and calculations

	Initial reading	Final reading	Vol ume of FAS
Blank			
Sample			

(COD) mg/l =((A-B)*M *8000) / (ml of Sample)

A=ml of FAS consumed for blank

B= ml of FAS consumed for sample

M= Molarity of FAS

(COD) mg/l = (A-B)*M *8000 / (ml of Sample)

A=ml of FAS consumed for blank

B= ml of FAS consumed for sample

M= Molarity of FAS

Results

The given sample contains the COD value of _____

Comments

According to central pollution control Board standards, the concentration of COD in waste water should be limited to 250mg/l

Environmental significance

- COD is used for measuring the organic strength of domestic waste.
- COD is used for measuring the organic matter in municipal and industrial waste
- The test provides accurate results within a short period.
- COD helps in determining the theoretical oxygen demand of organic matter.

Experiment 14: Air Quality Monitoring (Ambient, stack monitoring, Indoor air pollution)

Aim To understand operation of air quality monitor and compare air quality (CO, CO2, temperature, relative humidity) of indoor and outdoor air environments.

Background: Indoor Air Quality monitoring: 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.

Description of the instrument: The ambient air conditions measuring instrument, for assessing Indoor Air Quality and tuning and testing VAC systems, stands out on account of its efficient measurement process. The user-friendly measuring instrument has the right measurement engineering for every application and different flow speeds; for example, thermal probes, vane or Pitot tube measurement. Probe-controlled menus and selectable user profiles, e.g. for duct measurement or long-term measurement, ensure the user friendly operation possible. The new IAQ probe measures Indoor Air Quality by measuring the CO_2 level, air moisture and air temperature. In addition, lux and comfort level probes can be connected to measure draught air. Clear analysis and archiving ensure convenient documentation. Temperature and humidity measurement is built-in in the new thermal probe. The special flow protocol professionally documents duct measurements. It is also possible to connect additional temperature and humidity probes. Readings from up to temperature or humidity probes can be displayed in the measuring instrument; data transmission is by radio, i.e. wireless.

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Instrument memory for 10,000 readings

PC software for analysis, filing and documentation of measurement data

Thermal probes, vane measurement and built-in differential pressure probe probe for Pitot tube measurement

IAQ probe, lux probe and comfort level probe

Procedure: Measure air quality of three indoor locations and one outdoor location and compare their air quality parameters.

Steps: For a given location:

1. Prepare a sampling assembly.

2. Set the time constant depending upon the required averaging period (Instrument can be switch on and it will display concentration).

3. Simultaneously instrument will start recording the concentration values in the memory card. Using data transfer cable (i.e. RS232 cable) can download data from instrument to personal computers.

Experiment 14: Determination of Sound by Sound level meter at different location

Aim: To measure equivalent sound level and intensity of sound at various locations

Apparatus: Sound level meter

Principle: Sound pressure coming from a source converted to a small current of electricity, the electrical signals thus produced are amplified. In electronic circuitry called a weighing a readout built into the meter so as to produce a readout that closely resembles a human response.

Theory: Sound is the physiological sense resulting from pressure vibration in the air which acts on the surface of ear drum. The ear converts this pressure vibration into electrical signals which are then interpreted by as sound. Sound produced by all vibrating bodies is not audible. The number of vibrations pre second i.e the frequency should be greater than 20Hz (infrasonic) and less than 20,000 Hz (Ultrasonic). It is called as the frequency limits of audibility. Unwanted sound is called noise. An understanding of the fundamentals of sound generation and propagation is essential to the engine responsible for initiating and carrying out noise control programmes.

Equipment Description: A sound level meter consists of a microphone, coupled to good amplifiers and terminating in a logarithmic indicator calibrated in decibels. A sound meter, however, incorporates "frequency weighted" network designated to provide a closer relationship between the sensitivity of instrument and the response of human ear. A series of there internationally accepted weighing scales has been adopted. These are the A, B and C weighting networks. The A-network approximates human response to low-intensity sounds, the B-network approximates the response to medium-intensity sounds, and the C-network approximates the human response to high-intensity sounds. Since the readings are not actually the true sound pressure levels, readouts from the A, B and C networks are called sound levels, not sound pressure levels.

Procedure:
- Slide the power on/off switch to the on position
- Select the proper range of sound level in dB on the range switch
- Hold the sound level meter and face the noise sensor towards the noise source.
- Note down the noise levels at various locations
- Calculate the equivalent noise level by using the following formula

$$Leq = 10 \log [\Sigma(10)Li/10 * ti]$$

Plotting the graph of sound level v/s time represents the fluctuating sound with its equivalent energy level.

Application:

Noise level meters are used to measure noise in commercial, industrial and residential applications. Local planning commission prior to approval of construction and building permits use noise levels. Sound level meters are often used for projects to prove compliance with local ordinance. Other applications include factory, office and machinery sound level measurements, traffic and noise reduction studies and many more.

Observations & Calculations:

Table 1: Sound levels at different locations

Sl.No.	Location	Measured noise level in dB			Average (dB)
		1	2	3	
1					
2					
3					
4					

Equivalent Noise Level (Leq)

Leq is defined as the constant noise level, which, over a given time, expands the same amount of energy, as is expanded by the fluctuating levels over the same time. This value is expressed by the equation

Leq=10 log{$\Sigma(10)Li/10*ti$} .... (dB)

Where,

N = Total number of sound samples

Li = The noise level of any ith sample

ti = Time duration of ith sample, expressed as fraction of total sample time.

For equivalent sound level (Leq)

Total time =_____minutes   Time of interval =_____minutes

Location :_____

Time (min)	Measured Noise Level (dB)				Vehicles			
	1	2	3	Avg	2	3	4	>4

Results:

Comments:

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BUREAU OF INDIAN STANDARDS (BIS) SPECIFICATION FOR

DRINKING WATER (BIS: 10500 — 1991)

Characteristic or parameter	DL	Undesirable effect outside the Desirable Limit	PL
Color, Hazen units, max	05	Above 05 consumer acceptance decreases.	25
Odor (TON)	Un-objectionable	-	-
Taste	Agreeable	-	-
Turbidity, NTU, max	05	Above 05, consumer acceptance decreases	10
pH value	6.5 - 8.5	Beyond this range the water will affect the mucous membrane and I or water supply system.	No relaxation
Total hardness (as $CaCO_3$) mg/L, max	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 I appearances are affected, has adverse affect on domestic uses and water supply structures, and promotes iron Bacteria.	1.0
Chlorides (as Cl) mg/L, max	250	Beyond this limit, taste, corrosion and palatability are affected.	1000
Residual, free chlorine, mg/L, mm	0.2	-	-
Total Dissolved Solids (TDS) 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

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DL = Desirable Limit also known as requirable limit.

PL = Permissible Limit in absence of alternate source i.e., if the river dries up during summer season, the water demand has to be met by underground resources

Characteristic or parameter	DL	Undesirable effect outside the Desirable Limit	PL
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
Sulfate (as SO_4) mg/L, max	200	Beyond this causes gastro intestinal irritation When magnesium or sodium are present	400
Nitrate (as NO_3) mg/L, max	45	Beyond this Mathemoglobinemia (blue baby disease) 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
Phenotic compounds (as C_5H_5OH) 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
Cadmium (as Cd) mg/L, max	0.01	Beyond this, the water becomes toxic	No relaxation
Selenium (as Se)	0.01	Beyond this, the water becomes toxic	No

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mg/L, max			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

Characteristic or parameter	DL	Undesirable effect outside the Desirable Limit	PL
Polynuclear Aromatic Hydrocarbons (as PAH) mg/L, max	-	May be carcinogenic	-
Mineral oil, mg/L, Max	0.01	Beyond this limit undesirable taste and odor after chlorination takes place.	0.03
Pesticides, mg/L, max	Absent	Toxic	0.001
Radioactive			

materials: a) α (Alpha) emitters, Bq/L, max	-	-	0.1
b) β (Beta) emitters, pci/L, max	-	-	1
Alkalinity, mg/L, Max	200	Beyond this limit taste becomes unpleasant	600
Aluminium, (as Al), mg/L, max	0.03	Cumulative effect is reported to cause dementia	0.2
Boron, (as Bo), mg/L, max	01	-	05
Magnesium, (as Mg), mg/L, max	30	Encrustation to water supply structure and adverse effects on domestic use.	100
Chromium (as 0.05 Cr6) mg/L, max	0.05	May be carcinogenic above this limit	No Relaxation
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

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TREATED EFFLUENT STANDARDS

GENERAL STANDARDS FOR DISCHARGE OF ENVIRONMENTAL

POLLUTANTS: SEWAGE (AS PER CPCB, NEW DELHI)

Characteristics/ Parameter	STANDARDS			
	Inland surface water	**Public Sewers**	**Land for Irrigation**	**Marine coastal areas**
Color and odor	*	-	*	*
Suspended solids mg/L, max.	100	600	200	a) For process waste water- 100 b) For cooling water effluent 10% above total suspended matter of effluent
Particle sizes of Suspended solids	Shall pass 850 micron IS sieve	-	-	a) Floatable solids, Max. 3mm size b) Settelable solids, max, 850 microns
pH value	5.5 - 9.0	5.5 - 9.0	5.5 - 9.0	5.5 - 9.0
Temperature in °C	Shall not exceed 5° C	-	-	Shall not exceed 5° C

	above the receiving water Temp			above the receiving water Temp
Oil and grease, mg/L, max.	10	20	10	20
Total residual chlorine, mg/L, max	1.0	-	-	1.0

* = All efforts should be made to remove color & odor as far as practicable.

Note: Inland surface water - Streams, Rivers, Ponds, Lakes etc.

Marine coastal areas — in to sea or ocean.

Characteristics/ Parameter	STANDARDS			
	Inland surface water	Public Sewers	Land for Irrigation	Marine coastal areas
Free ammonia as (NH3) mg/L, max	5.0	-	-	5.0
Biochemical Oxygen Demand, mg/L max. (BOD_5 or BOD_3)	30	350	100	100
Chemical Oxygen Demand (COD) mg/L	250	-	-	250
Arsenic (as As),	0.2	0.2	0.2	0.2

mg/L, max				
Mercury (as Hg) mg/L, max	0.01	0.01	-	0.01
Lead (as Pb) mg/L, max	0.1	1.0	-	2.0
Calcium (as Ca) mg/L, max	2.0	1.0	-	2.0
Hexavalent Chromium s 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
Dissolved Phosphate (as P) mg/L, max	5.0	-	-	-

Characteristics/ Parameter	STANDARDS			
	Inland surface water	Public Sewers	Land for Irrigation	Marine coastal areas
Manganese (as Mn) mg/L	02	02	-	02
Iron (as Fe) mg/L	03	03	-	03

Sulfide (as S) mg/L, max	2.0	-	-	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
Vanadium (as V) mg/L	0.2	0.2	-	0.2
Nitrate Nitrogen mg/L	10	-	-	20
Ammonical nitrogen (as N), mg/L, max.	50	50	-	50
Total kjeldhal nitrogen [N], mg/L, max	100	-	-	100
Radioactive materials: a) Alpha emitters α [micron curie/mL] max b) Beta emitters β [micro curie fmL] max	10^{-7} 10^{-6}	10^{-7} 10^{-6}	10^{-8} 10^{-7}	10^{-7} 10^{-6}

Source: Sathpal Puliani & B. Shivalingaiah, "The Environment Protection and Pollution Control Manuar Karnataka Law Journal publications, 2000 Edition, Law publishers, Bangalore.

CPCB: Central Pollution Control Board. New Delhi.

DOs and DON'Ts in the Laboratory

11. Do thoroughly clean the glassware before and after use.

12. Do handle the glassware carefully.

13. Do not handle chemicals with bare hands.

14. Do not blow out the last drop from the pipette. When the liquid has drained out completely, touch the tip of the pipette to the inner surface of the vessel.

15. Do not add water to acids. Do always add acid to water.

16. Do use large volumes of water, when a person is splashed with acid to prevent serious burns.

17. Do weigh the articles in a balance only at room temperature.

18. . Do use different pipette for different reagents

19. Do not pipette out acids and other toxic reagents by mouth.

20. Do read the level of the curve (meniscus), in all volumetric glassware, with the eye at approximately the same level as the curve of solution.

General Information

In water and wastewater analysis, the results are usually reported in terms of mg/L of some particular ion, element or compound. It is most convenient to have the standard titrating agent of such strength, that 1mL is equivalent to 1mg of material being measured. Thus 1 litre of the standard solution is usually equivalent to 1g of the standard substance.

Normality

The desired normality of the titrant is obtained by the relationship of 1 to the equivalent weight of the measured material. Thus normality of acid solution to measure ammonia, ammonia nitrogen, and alkalinity as $CaCO_3$

Ammonia — 1/eq. wt. = 1/17 = N/17 = 0.0588N

Ammonia N — 1/eq. wt. = 1/14 = N/14 = 0.0715N

Total Solids = $\dfrac{W_2-W_1 \times 10^6}{\text{ml of sample}}$ mg/L

Total Solids = $\dfrac{W_2-W_1 \times 10^6}{\text{ml of sample}}$ mg/L

The normality of basic solution to measure mineral acidity as $CaCO_3$ is:

Acidity — 1/eq. wt. = 1/50 = N/50 = 0.020N

 The normality of silver nitrate to measure chloride and sodium chloride is:

Chloride — 1/eq. wt. = 1/35.45 = N/35.45 = 0.0282N

Sodium chloride — 1/eq. wt. = 1/58.44 = N/58.44 = 0.071N

Thus the substance measured is calculated as follows:

= ml of titrant used × 1,000 mg / L

      ml of sample

Most materials subjected to the analysis of water and wastewater fall in the realm of dilute solutions i.e., a few mg in a litre. So the results are normally expressed in mg/L or ppm. Parts per million (ppm) is a weight ratio; but mg/L is a weight by volume ratio. The relationship is given as follows:

ppm = mg / L

If concentrations are less than 0.1 mg /L, express them in µg/L (micrograms per litre).

      If concentrations are more than 10,000 mg/L, they are expressed in percentages

Lab Safety:

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.

Lab Etiquette:

Return all chemicals and supplies to the proper location after use. It is necessary to take chemicals from reagent bottles, pour out slightly more than the amount of chemical needed into a clean beaker. Never pour a chemical back into a reagent bottle. Clean up for the next person. At the conclusion of each work period, all used glassware must be cleaned and set to drain. Remove label tape, scrub inside of glassware with water and laboratory detergent, rinse with tap water, rinse with distilled water, and place cleaned

glassware on a rack to dry. No experiment is complete until the laboratory is cleaned.

Procedure for cleaning of glassware in laboratory

Glassware cleaning

1. Clean the equipment thoroughly with soap and water for basic cleaning. You may need to use a wire brush to remove some residue. Detergent using bottle brushes and scouring pads can be used as needed.

2. After cleaning, rinse the glassware with running tap water. When test tubes, graduates, flasks and similar containers are rinsed with tap water, allow the water to run into and over them for a

short time, then partly fill each piece with water.

3. Thoroughly shake and empty at least six times and ensure that all soap residue is removed.

Note:

• Do not use cleaning brushes that are so worn that the spine hits the glass. Serious scratches may result. Scratched glass is more prone to break during experiments. Any mark in the uniform surface of glassware is a potential breaking point, especially when the piece is heated. Do not allow acid to come into contact with a piece of glassware before the detergent (or soap) is thoroughly removed. If this happens, a film of grease may be formed.

• To prevent breakage when rinsing or washing pipettes, cylinders or burettes, be careful not to let

tips hit the sink or the water tap.

Sterilizing Contaminated Glassware

• Autoclave glassware or sterilize it in large steam ovens or similar apparatus. If viruses or spore bearing bacteria are present, autoclaving is absolutely necessary.

Handling and Storing

• Protect clean glassware from dust. This is done best by plugging with cotton, corking, taping a heavy piece of paper over the mouth or placing the glassware in a dust-free cabinet.

• Store glassware in specially designed racks. Avoid breakage by keeping pieces separated.

Meaning of Water Quality Analysis

Water quality standards are put in place to ensure the efficient use of water for a designated purpose. Water quality analysis is to measure the required parameters of water, following standard methods, to check whether they are in accordance with the standard.

Requirement of Water Quality Analysis

Water quality analysis is required mainly for monitoring purpose. Some importance of such assessment includes: 1. To check whether the water quality is in compliance with the standards, and hence, suitable or not for the designated use. 2. To monitor the efficiency of a system, working for water quality maintenance. 3. To check whether up gradation / change of an existing system is required and to decide what changes should take place. 4. To monitor whether water quality is in compliance with rules and regulations.

Sampling of Water for Analysis

A common cause of error in water quality analysis is improper sampling. The results of a water quality analysis of a sample show only what is in the sample. For the results to be meaningful, the sample must be representative i.e., it must contain essentially the same constituents as the body of water from which it was taken. The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled. The complexity of water quality as a subject is reflected in the many types of measurements of water quality indicators. The most accurate measurements of water quality are made on-site, because water exists in equilibrium with its surroundings. Measurements commonly made on-site and in direct contact with the water source in question include temperature,' pH, dissolved oxygen, electric conductivity, etc. More complex measurements are often made in a laboratory requiring a water sample to be collected, preserved, transported, and analyzed at another location.

Requirements for Sampling

• Meet the requirements of the sampling program. • Handle the sample carefully so that it does not deteriorate or become contaminated or compromised before it is analyzed. •

Ensure sampling all equipment are clean and quality assured before use. • Use sample containers that are clean and free of contaminants. • Rinse the bag/bottle at least twice with the sample water prior to filling and closing. • Fill bag/bottle as full as possible. Half-filling leaves more room for oxygen which will promote degradation of your sample. • If sampling a body of running water, point the mouth of the bag upstream and your hands downstream to avoid contamination. • If sampling from a water faucet, run the faucet for 1 minute before obtaining a sample. • Make records of every sample collected and identify every bottle e.g., take notes and photographs, fill out tags, etc. • Place the sample into appropriate, labeled containers. • All samples must be preserved as soon as practically possible.

Sample Collection bottles, Size and Materials

The methods that will be followed will determine the type of bottles used. For example, samples for metals' analyses are usually collected in plastic bottles, while analyses for volatile organics and pesticides are collected in glass containers. Bottles used to collect samples for bacteria should be sterilized. Certain analysis like volatile organics and radon require vials that are to be filled leaving no head space, which keeps these analytes dissolved in the water, preventing them from escaping into the air. Additionally, some analyses require samples to be collected in amber colored bottles. These darker bottles are for analytes that break-down in sunlight, which helps keep these contaminants from breaking down while in transit to the laboratory for analysis. The size of the container is important to ensure enough samples to run the analysis needed.

Water Sampling Techniques: Water sampling can be done in any of the following three methods depending on test requirements: • Grab sampling • Composite sampling • Integrated sampling

Grab sampling: Grab Samples are samples collected at a particular time and space. They represent the composition at that time and place. When a source is known to vary in time e.g.in case of waste effluents, grab samples collected at suitable time intervals and analyzed separately.

Composite sampling: Composite samples are a mixture of grab samples collected at one sampling point at different times. The composite samples are useful for observing values. Individual samples are collected in wide mouth bottles every hour and mixed in volume proportional to the flow or by using specially designed automatic sampling devices.

Advantages of Composite Samples:

- reduced costs of analyzing a large number of samples.

- More representative samples of varied conditions.

- Larger sample sizes when amounts of test samples are limited.

Disadvantages of Composite Samples:

- Loss of analytic relationships in individual samples.

- Potential dilution of any parameter below detection levels.

- increased potential analytical interferences.

 - increased possibility of analytic interactions.

Integrated sampling: Integrated samples are a mixture of grab samples collected from different points simultaneously and mixed in equal volumes.

Surface Water Sampling Techniques:

When the water source is accessible

Rinse the sampling vessel with water on site 3-4 times. Care must be taken to avoid contaminating water to be sampled during rinsing. - Submerge the sampling vessel gently, fill it with the water sample and close it tightly. If the collected water sample may be frozen, leave some space for expansion equivalent to about 10% of the sampling vessel.

When the water source is inaccessible-

A rope 'attached to the bucket are often used. Scoops with adjustable shafts are convenient. Items made of synthetic resins such as polypropylene can also be used.

Ground Water (from well) Sampling Techniques

A bailer in is a hollow tube used to retrieve groundwater samples from monitoring wells. Bailers are tied to a piece of rope or a piece of wire and lowered into the water column. Once lowered, the bailer uses a simple ball check valve to seal at the bottom in order to pull up a sample of the groundwater table.

Important Viva-voce questions for reference to Students

1. pH is one of the most important controlling factors for treatment and chemical analysis of water and wastewater explain.

2. Define pH in terms of hydrogen-ion (H^+) concentration and hydroxyl-ion (OH-) concentration. An increase in pH of one unit represents how much decrease in hydrogen ion concentration?

3. What is meant pH of a solution?

4. What is meant by buffer solution?

5. Can you explain the pH scale briefly?

6. What is the standard limit of pH in the drinking water?

7. Gives the examples of the solution of the acidity and alkalinity

8. What is the electrode used in the determination of the pH?

9. Discuss the importance of alkalinity in water for different treatment processes.

10. Define total alkalinity, phenolphthalein alkalinity, and methyl orange alkalinity. Discuss their dominant pH range.

11. How does pH play a role in affecting alkalinity and acidity of given water sample?

12. What is meant by acidity? What are the effects of the acidity?

13. Explain the principle involved in the determination of acidity?

14. What are the treatment techniques adopted for the reduction of acidity?

15. What is meant by acidity? What are the effects of the acidity?

16. Explain the principle involved in the determination of acidity?

17. What is the treatment techniques adopted for the reduction of acidity?

18. What are the effects of acidity in building construction activity/ concrete?

19. What are the principle ions responsible for the cause of alkalinity?

20. Explain the effects of alkalinity on water bodies?

21. How alkalinity does affects on plants?

22. What is meant by hardness of water?

23. What are the different types of hard water?

24. What are the principal anions and cations responsible for causing of hardness of water?

25. What are the effects of the hard water in domestic as well as the industrial usages?

26. What is treatment methods adopted for removing the hardness of water?

27. Explain the principle for the determination of the hardness using EDTA?

28. What is the standard limit of the hard water?

29. If the ground water is found to be saltier in taste then what sort of conclusion you come across it?

30. Among finished drinking water, raw wastewater and de-ionized water, which water is expected to have the highest carbonate hardness and why?

31. What is the limit for the Dissolved oxygen present in the water?

32. What are the factors affecting the D.O?

33. How can you determine the presence or absence of DO in the given samples?

34. How can you relate the DO with BOD in what way it can be used to assess in wastewater treatment.

35. How does the DO Concentration varies in lakes and rivers?

36. Why do you take 203 ml of the sample from the BOD Bottle?

37. What is the difference between seeded and unseeded BOD samples?

38. What happens if the Sodium chloride concentration increases in the uptake of plants?

39. What are the different forms of chlorination?

40. What is the difference between disinfection and sterilization?

41. Justify how can you treat the water contaminated with the microorganisms at home?

42. Define available chlorine and residual chlorine and chlorine demand

43. What is the chemical name of bleaching powder? And how much bleaching powder concentration must be present in the treated water?

44. What is the permissible and tolerable total dissolved solids in drinking water as per Indian standards?

45. What are the different types of solids that you come across in your laboratory?

46. How the turbidity does affect the disinfection process?

47. Explain the principle in the determination of the turbidity.

48. Explain how turbidity of the water is caused?

49. Mention the standards for the turbidity

50. What are the differences between BOD and COD?

51. What is the permissible limit of COD for inland sewers and surface water bodies?