

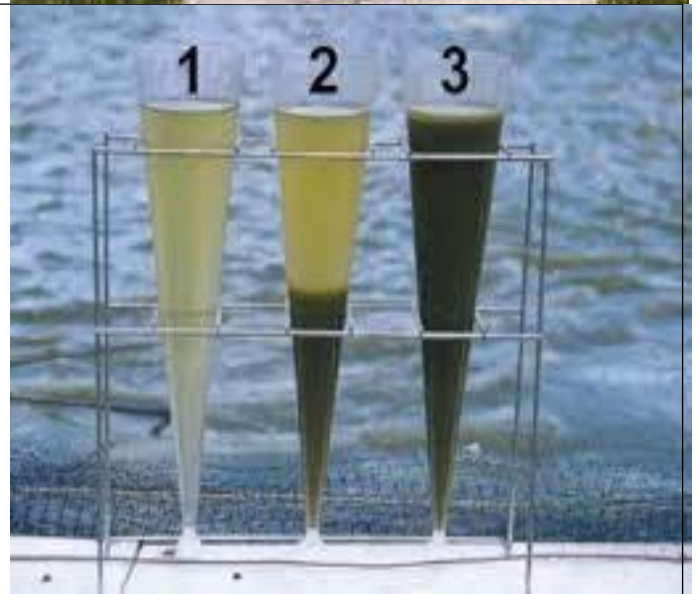


SRM VALLIAMMAI ENGINEERING COLLEGE
(An Autonomous Institution)



S.R.M NAGAR, KATTANKULATHUR - 603203.

1903610 - WATER AND WASTE WATER ANALYSIS
LABORATORY MANUAL (SIXTH SEMESTER)



DEPARTMENT OF CIVIL ENGINEERING
REGULATION 2019
2021-2022

PREFACE

This instruction manual has been prepared by the DEPARTMENT OF CIVIL ENGINEERING to facilitate instruction during practical classes and further to be used as a reference manual by the Civil Engineering students of this college. This manual covers explanation of experiments included in the syllabus as per Autonomous Regulation 2019 in Environmental Engineering Laboratory for the B.E Degree Course. Any suggestions for the improvement of the manual will be gratefully received.

Prepared by

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REGULATION 2019

1903610 WATER AND WASTE WATER ANALYSIS LABORATORY

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COURSE OBJECTIVES:

- To analyse the physical, chemical and biological characteristics of water and wastewater.
- To quantify the dosage requirement for coagulation process.
- To study the growth of micro-organism and its quantification.
- To study about the oxygen demand in water.
- To gain knowledge on water pollution and the treatment methods.

Course Content:

1. Physical, Chemical and biological characteristics of water and wastewater
2. Jar test
3. Chlorine demand and residual test
4. Growth of micro-organism

COURSE OUTCOME:

On the completion of the course, the students will be able to:

- Quantify the pollutant concentration in water and wastewater.
- Suggest the type of treatment required and amount of dosage required for the treatment.
- Examine the conditions for the growth of micro-organisms
- Understand the impact of water and waste water treatment on people and environment.
- Understand and apply ethical issues associated with water.

TOTAL: 60 PERIODS

List of Experiments:

1. Determination of pH, Turbidity and conductivity in water.
2. Determination of Hardness in water.
3. Determination of Alkalinity and Acidity in water sample.
4. Determination of Chlorides in given samples.
5. Determination of Phosphates and Sulphates in given water samples.
6. Determination of Optimum Coagulant dosage in waste water sample.
7. Determination of residual chlorine and available chlorine in bleaching powder.
8. Determination of suspended, settleable, volatile and fixed solids in waste water.
9. Determination of Dissolved Oxygen and BOD for the given sample.
10. Determination of COD for given sample.
11. Determination of Sodium and Potassium for the given sample.
12. Determination of SVI of Biological sludge and microscopic examination.
13. Determination of MPN index of given water sample.

LABORATORY SAFETY PROCEDURES

DO'S

- Wear Mask and follow the COVID precautionary measures.
- Know the potential hazards of the materials used in the laboratory.
- Wear personal protective apparel when working with chemicals. This includes eye protection, lab coat, gloves.
- Wash skin promptly if contacted by any chemical, regardless of corrosivity or toxicity.
- Shoes must cover the entire foot. Open toed shoes and sandals are inappropriate footwear in laboratories.
- Restrain and confine long hair and loose clothing. Pony tails and scarves used to control hair must not present a loose tail that could catch fire or get caught in moving parts of machinery.
- Understand the Hazards of Your Chemicals - Read all labels.
- Read the level of the curve (Meniscus) in all volumetric glass coarse with the eye approximately the same level as the curve of the solution.
- Clean all the apparatus used with distilled water before and after the experiment.
- Have your Lab manual, Observation and record note for every lab classes.

DON'T

- Eat, drink, chew gum, or apply cosmetics in rooms or laboratories where chemicals are used or stored.
- Drink water from laboratory water sources.
- Smell chemicals, taste chemicals, or pipette by mouth.
- Put hazardous chemicals down the drain
- Use squirt/wash bottles over the sink
- Allow waste to accumulate.
- Add water to concentrated acid. A concentrated acid should be added carefully to water.

Basic Conversions:

Percent by weight: To make up a solution based on percentage by weight, one would simply determine what percentage was desired.

$$\text{Weight percentage} = (\text{Weight of solute} / \text{weight of solution}) \times 100\%$$

Percent by volume: Solutions based on percent by volume are calculated the same as for percent by weight, except that calculations are based on volume. Thus one would simply determine what percentage was desired

$$\text{Volume percentage} = (\text{Volume of solute} / \text{Volume of solution}) \times 100\%$$

Percent by weight/volume: Another variation on percentage concentration is weight/volume percent or mass/volume percent. This variation measures the amount of solute in grams but measures the amount of solution in milliliters.

$$\text{Weight/Volume percentage} = (\text{Weight of solute} / \text{Volume of solution}) \times 100\%$$

Dilution: Dilution is the mixing of a small accurately measured sample with a large volume of sterile water or normal saline called (diluent or dilution blank).

$$\text{Dilution} = \text{Volume of sample} / \text{total volume of (sample+ Diluent)}$$

$$\text{Dilution Factor} = \text{Total Volume of (Sample+ Diluent)} / \text{Volume of sample}$$

For drinking water the following standards should be strictly maintained.

S.NO	PARAMETER	MAXIMUM ALLOWABLE CONCENTRATION
1.	Colour	5 Units
2.	Taste	Unobjectionable
3.	Odour	Unobjectionable
4.	Turbidity	5 Units
5.	Total Solids	500 mg/l
6.	Carbonate Alkalinity	120 mg/l
7.	Chlorides	200 mg/l
8.	Residual Free Chlorine	0.20 mg/l
9.	BOD	Nil
10.	Iron	0.20 mg/l
11.	Lead	0.10 mg/l
12.	Selenium	0.01 mg/l
13.	Chromium	0.05 mg/l
14.	Cyanide	0.01 mg/l
15.	Nitrates	45 mg/l
16.	Copper	1.0 mg/l
17.	Zinc	5.0 mg/l
18.	Calcium	75 mg/l
19.	Magnesium	50 mg/l
20.	Sulphate	200 mg/l
21.	Phenolic Substances	0.001 mg/l
22.	Manganese	0.01 mg/l
23.	Cadmium	0.01 mg/l
24.	Silver	0.05 mg/l

25.	Barium	1.00 mg/l
26.	Mercury	0.001 mg/l
27.	Pesticides	Nil
28.	B-coli	Nil in 100 ml
29.	Most probable number(MPN)	1 in 100 ml

S.NO	PARAMETER	PERMISSIBLE RANGE OF CONCENTRATION
1.	pH	7 to 8.5
2.	Dissolved Oxygen	5 to 8.5 mg/l
3.	Hardness	75 to 115 mg/l
4.	Fluorides	0.8 to 1.0 mg/l

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Ex. No	Date	Name of the Experiments	Page No	Staff Signature
1		A. Determination of pH in given samples		
		B. Determination of Turbidity in given samples		
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2		Determination of Hardness in given sample		
3		A. Determination of Alkalinity in given sample		
		B. Determination of Acidity in given sample		
4		Determination of Chlorides in given sample		
5		A. Determination of Phosphates in given sample		
		B. Determination of Sulphates in given sample		
6		Determination of Optimum Coagulant dosage in given sample		
7		A. Determination of residual chlorine in bleaching powder		
		B. Determination of available chlorine in bleaching powder		
8		Determination of suspended, settleable, volatile and fixed solids in given sample		
9		Determination of Dissolved Oxygen and BOD for the given sample		
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		Determination of oil and grease in given wastewater sample		
		Determination of Calcium in the given wastewater sample		

Ex.No:1

Date: **Determination of pH by using pH meter**

Aim:

To find the pH present in the given water sample.

Apparatus Required:

pH meter, Electrode, Buffer solutions, filter papers etc.

Principle:

pH refers to the hydrogen ion activity and is expressed as the logarithm of reciprocal of hydrogen ion activity in molar per litre. It can be measured by calorimetric methods using various indicators or electrometrically by using pH meter employing hydrogen ion-sensitive electrodes. The basic principle of electrometric pH measurement is determination of activity of the hydrogen ion by potentiometric measurement using a standard hydrogen electrode and a reference electrode.

Interference:

pH measurements are affected by temperature. In the pH meter, provision is made for setting the instrument to room temperature before taking the pH readings. The Buffer solutions used for standardization must be kept in the refrigerator. Always report the temperature at which pH is measured.

Procedure:

1. Connect the pH electrode to the pH meter input terminal. Adjust the manual temperature setting to room temperature.
2. Wash the pH electrode with distilled water and wipe dry using filter paper. Insert the pH electrode into a known buffer of 7.0 pH. If it is different from 7.0 pH, adjust the reading to 7.0 pH by varying the AP control.
3. Wash the electrode with distilled water and wipe dry. Insert the electrode into another known buffer, 4.0 pH. If it is different from 4.0 pH, adjust the reading to 4.0 pH.
4. Wash the electrode with distilled water and wipe dry. Repeat the procedure with 7.0 pH and 4.0 pH until consecutive correct readings are obtained.
5. Now the pH electrode and the pH meter have been matched calibrated.
6. Wash the electrode with distilled water and wipe dry. Insert the electrode into the unknown sample and take the displayed reading.

Result:

The pH of the given water sample is -----

Tabulation:

S.NO	SAMPLES	pH

Ex.No:1B

Date: **Determination of Turbidity in water sample**

Aim:-

To determine the turbidity of the given sample water by Nephelometric method.

Apparatus required: -

Nephelo turbidity meter.

Principle: -

Turbidity is a measure of the extent to which light is either absorbed or scattered by suspended material present in the water. Turbidity in surface waters results from the erosion of colloidal material such as clay, silt, rock fragments and metal oxides from soil, vegetable fibers and micro-organisms may also contribute to turbidity. Drinking water supplies requires special treatment by chemical coagulation and filtration before it may be used for public water supply.

This turbidity can be brought down to required level by adding coagulants. Coagulants when added to water it will form a gelatinous substance known as floc and this will arrest the fine suspended and colloidal particles. These arrested particles will settle down rapidly because of increase in their size.

Interference: -

Turbid waters are aesthetically displeasing and are not accepted for domestic use. The colloidal matter associated with turbidity provides adsorption sites for chemicals and biological organisms that may be harmful or cause undesirable tastes and odour. Disinfection of the turbid waters is difficult and unsatisfactory, since the colloids partially shield organisms from the disinfectant. This IS values for drinking water is 10 to 25 NTU.

Reagents: -

1. Turbidity free water: - Pass distilled water through a lower turbidity than distilled water, discard the first 200ml, collected. If filtration does not reduce turbidity use distilled water.
2. Stock turbidity solutions:
 - i) **Solution 1:** - Dissolve 1.0 grams hydrazine sulfate $(\text{NH}_2)_2 \cdot \text{H}_2\text{SO}_4$ in distilled water and dilute it to 100 ml in a makeup flask.
 - ii) **Solution 2:** - Dissolve 10.0 grams hexamethylene tetramine $(\text{CH}_2)_6\text{N}_4$ in distilled water and dilute it to 100ml.
 - iii) **Solution 3:** - In a 100ml flask, mix 5ml. each of solution 1 and 2. Allow it to stand 24 hours, then dilute it to 100ml and mix thoroughly. The turbidity of this solution is 400 NTU.
 - iv) **Standard Turbidity Solution:** - Take 10.0ml of solution 3 in a 100ml make up flask and dilute it to 100ml with turbid free water. The turbidity of this suspension is 40 NTU.

PROCEDURE:-

a) Calibration of Nephelometer:-

- i) Select proper range of NTU on Nephelometer.
- ii) By placing distilled water in Nephelometer test tube, set the Nephelometer reading to zero by using the knobs provided for zero setting.
- iii) Using the standard turbid solution (i.e. 40 NTU), calibrate the Nephelometer (i.e. adjust the Nephelometer reading to 40 NTU using calibration knob).

OBSERVATIONS:-

S.No	SAMPLE	NTU

b) Determination of turbidity of sample water:

i) For samples having turbidities less than 40 NTU: Thoroughly shake the sample so as to remove any air bubbles and pour it into meter cell. Read out the turbidity of the sample from the digital display.

ii) For samples having turbidities above 40 NTU: Dilute sample with 1,2 or 3 volumes of turbidity free water and convert the value obtained as below.

If five volumes of turbidity free water were added to one volume of sample and the diluted sample showed a turbidity of 30 NTU, then the actual value is equal to 180 units. i.e.

$$\text{Nephelometric turbidity units (NTU)} = \frac{A(B + C)}{C}$$

Where

A = Turbidity found in diluted sample, B = Volume of dilution water in ml

C = Sample volume for dilution in ml.

RESULTS: -

Ex.No:1C

Date: **Determination of Conductivity in water sample**

Aim:

To determine the Conductivity of the given sample

Apparatus required:

Conductivity Meter, Flask, Beaker, Wash Bottle

Principle:

Conductivity is the capacity of water to carry an electrical current and varies both with number and types of ions the solution contains, which in turn is related to the concentration of ionized substances in the water. Most dissolved inorganic substances in water are in the ionized form and hence contribute to conductance. Conductivity measurement gives rapid and practical estimate of the variations in the dissolved contents of water.

Environmental Significance:

Electrical conductivity measurements are often employed to monitor desalination plants. It is useful for detection of impurities in water. Used for quantitative measurement of ionic constituents dissolved in water, which are important for boiler feed water and cooling water etc.; Used for checking correctness of water analysis as there is a distinct relationship between conductivity and total dissolved solids (TDS). Conductivity data is useful in determining the suitability of water and wastewater for disposal on land. Irrigation waters up to 2 millisiemens/cm conductance have been found to be suitable for irrigation depending on soils and climatic characteristics.

Reagents:

0.01 N, KCL solution: Dissolve 0.746 grams of potassium chloride in 1 litre of distilled water.

Procedure:

1. Rinse the electrode thoroughly blot and dry.
2. Immerse the electrode in 0.01 N KCL solution and the conductivity should be 1413 micro mhos at 25°C Temperature.
3. Now the instrument is calibrated.
4. Remove the cell from KCL solution and wash it thoroughly with distilled water.
5. Immerse the electrode/cell in the unknown sample whose conductivity is to be determined.
6. Note down the instrument reading. If the cell constant is given on the cell itself, then the measured value of conductivity = Cell Constant C X Screen reading

Result:

Tabulation:

Sl.No	Sample	Conductivity(mS/cm)	Temperature(°C)

Ex. No:2

Date: **Determination of Hardness in water sample**

Aim:

To determine the total hardness present in the given water sample.

Apparatus Required:

Burette, Conical flask, Pipette, Measuring jar.

Principle:

Hardness is generally caused by the Calcium and Magnesium ions present in water. Polyvalent ions of some other metals like Strontium, Iron, Aluminium, Zinc and Manganese are also capable of precipitating soap and thus contributing to hardness. However, the concentration of these ions is very low in natural waters, therefore hardness is generally measured as concentration of only Calcium and Magnesium (as Calcium Carbonate), which are far higher in quantity over other hardness producing ions. Calcium and Magnesium form a complex of wine red colour with Eriochrome Black-T at pH of 10.0 ± 0.1 . The EDTA has got a stronger affinity to Ca^{2+} and Mg^{2+} and therefore by EDTA, the former complex is broken down and a new complex of blue colour is formed.

Reagents:

1. EDTA solution 0.01N (Ethylene Diamine tetra Acetic Acid)
2. Buffer solution
3. Eriochrome Black-T Indicator.

Procedure:

1. Take 50ml water sample in a conical flask. If the sample is having higher Calcium, take a small volume and dilute to 50ml.
2. Add 1 ml of buffer solution.
3. If the sample is having higher amount of heavy metals add 1 ml of Na_2S solution.
4. Add 100-200 mg of Eriochrome Black-T indicator, the solution turns wine red.
5. Titrate the contents against EDTA solution. The end point colour changes from wine red to blue. Repeat the titrations to get concurrent values.

Result:

The total hardness present in the given water sample is -----

Inference:

The water containing 0 to 60 mg/L (milligrams per liter) as calcium carbonate is classified as soft; 61 to 120 mg/L as moderately hard; 121 to 180 mg/L as hard; and more than 180 mg/L as very hard.

Tabulation:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of EDTA (ml)
		Initial Reading	Final Reading		

Calculation:

$$\text{Hardness in mg/l of CaCO}_3 = \frac{\text{Volume of EDTA used} \times 1000}{\text{Volume of Sample}}$$

Ex.No:3A**Date:** **Determination of Acidity in water sample****Aim:**

To determine the acidity present in water & Wastewater sample

Theory:

Acidity of water is its quantitative capacity to neutralize a strong base to a designated pH. Acidity of water is significant in many water supply systems, because acidity influences certain chemical and biological process in water. Acidity of water is due to Hydrogen ions (H⁺) present in a sample of water. As a result, dissociation of Hydrogen ions is neutralized by titration with standard solution of a strong base in presence of an indicator. Dissolved Carbon dioxide is usually the major acidic component of surface water. Acidity is usually determined by titration with 0.02N solution of Sodium Hydroxide. The amount of Sodium Hydroxide required for the sample to reach the pH 4.5 in a measure of Methyl orange acidity and amount to reach pH 8.3 is a measure of total acidity.

Environmental Significance:

- Acidity interferes in the treatment of water. Carbon dioxide is of important considerations in determining whether removal by aeration or simple neutralization with lime / lime soda ash or NaOH will be chosen as the water treatment method.
- The size of the equipment, chemical requirements, storage spaces and cost of the treatment all depends on the carbon dioxide present.
- Aquatic life is affected by high water acidity. The organisms present are prone to death with low pH of water.
- High acidity water is not used for construction purposes. Especially in reinforced concrete construction due to the corrosive nature of high acidity water.
- Water containing mineral acidity is not fit for drinking purposes.
- Industrial wastewaters containing high mineral acidity is must be neutralized before they are subjected to biological treatment or direct discharge to water sources.

Apparatus:

Titration Stand with Burette, Conical Flask, Beaker, Measuring Cylinder, Pipette, Funnel, Wash Bottle etc

Reagents:

- 0.02 N NaOH Solution: Dissolve 0.8 grams of NaOH in 1 liter of distilled water.
- Phenolphthalein Indicator: Dissolve 0.5 gm of Phenolphthalein Indicator in 500 ml .95% of ethyl alcohol. Add 500 ml distilled water.
- Methyl orange Indicator: Dissolve 0.5 g of Methyl orange Indicator & dilute to 1000 ml with distilled water

Observation:

1. Titrant:
2. Indicators: Phenolphthalein and Methyl Orange Indicator
3. End Point:
 - a) Yellow to Faint Orange
 - b) Colorless to Faint Pink Color

Table: 1 Mineral Acidity: (Methyl Orange Indicator)

S.No	Volume of sample ml(V)	Burette Reading(ml)		Volume of NaOH (ml)(A)
		Initial	Final	

Table: 2 Phenolphthalein Acidity: (Phenolphthalein Indicator)

S.No	Volume of sample ml (V)	Burette Reading(ml)		Volume of NaOH (ml) (B)
		Initial	Final	

Calculations:

Mineral Acidity as CaCO_3 in mg/L = $\frac{\text{Volume of NaOH (A)} \times N \times 50 \times 1000}{V}$

V

Phenolphthalein Acidity as CaCO_3 in mg/L = $\frac{\text{Volume of NaOH (B)} \times N \times 50 \times 1000}{V}$

V

Total Acidity as CaCO_3 in mg/L = $\frac{\text{Volume of NaOH (A)} + \text{Volume of NaOH (B)} \times N \times 50 \times 1000}{\text{ml of sample}}$

ml of sample

Procedure:**A. Methyl Orange Acidity**

1. Take suitable volume of sample (25, 50 or 100 ml) in a Conical flask. (V)

2. Add 2 drops of methyl orange and see the colour. If color turns yellow, methyl orange acidity is absent. If colour turns orange, titrate with 0.02 N NaOH till colour changes to faint orange. Characteristic of pH 4.3-4.4. Note the volume of NaOH required (A).

B. Phenolphthalein Acidity

1. Take suitable volume of sample (25, 50 or 100 ml) in a Conical flask. (V)

2. Add 2-3 drops of phenolphthalein indicator, If the sample turns pinks color it means that, phenolphthalein acidity is absent and stop the experiment.

3. If there is no color change, titrate with 0.02N Standard NaOH solution till faint pink color appears indicating pH 8.3. The end point is colorless to faint pink color. Note additional volume of NaOH required (B).

Results:

Sample Used	Mineral Acidity (mg/L)	Phenolphthalein Acidity (mg/L)	Total Acidity(mg/L)

Inference:

Total Acidity/Phenolphthalein Acidity measures the neutralizing effects of essentially all the acid species present both strong and weak.

Methyl orange acidity primarily measures acidity due to dissolved CO₂ and other weak acids that are present.

Strong Mineral Acids contribute to the mineral acidity whereas weak acids such as carbonic acid, acetic acid present in the water contributes to Phenolphthalein Acidity.

Ex.No:3B**Date:** **Determination of Alkalinity in water sample****Aim:**

To determine the Alkalinity present in water sample

Theory:

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

Environmental Significance:

- Alkalinity is important for fish and aquatic life because it protects or buffers against rapid pH changes. Higher alkalinity levels in surface waters will buffer acid rain and other acid wastes and prevent pH changes that are harmful to aquatic life.

- Large amount of alkalinity imparts bitter taste in water.

- The principal objection of alkaline water is the reactions that can occur between alkalinity and certain in waters. The resultant precipitate can corrode pipes and other accessories of water distribution systems.

- Wastewaters containing excess caustic (hydroxide) alkalinity are not to be discharged into natural water bodies or sewers.

- Alkalinity as carbonate and bicarbonate of saline water is very important in tertiary recovery processes for recovering petroleum. Alkaline water offers better wetting to the formation rock and improve oil release. As an additional benefit, ions that provide alkalinity absorb on rock surfaces occupying adsorption sites and decrease the loss of recovery chemical by adsorption.

- The alkalinity value is necessary in the calculation of carbonate scaling tendencies of saline waters.

- The alkalinity acts as a pH buffer in coagulation and lime-soda softening of water.

- In wastewater treatment, alkalinity is an important parameter in determining the amenability of wastes to the treatment process and control of processes such as anaerobic digestion, where bicarbonate alkalinity, and any fraction contributed by volatile acid salts become considerations.

Apparatus:

Titration Stand with Burette, Conical Flask, Beaker, Measuring Cylinder, Pipette, Funnel, Wash Bottle etc.

Observation:

1. Titrant :
2. Indicators : Phenolphthalein and Methyl Orange Indicator
3. End Point : a) Pink to Colourless
b) Yellow to Faint Orange Color

Table: 1 Phenolphthalein Alkalinity: (Phenolphthalein Indicator)

S.No	Volume of sample ml (V)	Burette Reading(ml)		Volume of H ₂ SO ₄ (ml) (A)
		Initial	Final	

Table: 2 Methyl orange Alkalinity: (Methyl orange Indicator)

S.No	Volume of sample ml (V)	Burette Reading(ml)		Volume of H ₂ SO ₄ (ml) (B)
		Initial	Final	

Calculations:

$$\text{Phenolphthalein Alkalinity as CaCO}_3 \text{ in mg/L} = \frac{\text{Volume of H}_2\text{SO}_4 \text{ (A) } \times \text{N} \times 50 \times 1000}{V}$$

$$\text{Methyl orange Alkalinity as CaCO}_3 \text{ in mg/L} = \frac{\text{Volume of H}_2\text{SO}_4 \text{ (B) } \times \text{N} \times 50 \times 1000}{V}$$

$$\text{Total Alkalinity as CaCO}_3 \text{ in mg/L} = \frac{\text{Volume of H}_2\text{SO}_4 \text{ (A) + (B) } \times \text{N} \times 50 \times 1000}{V}$$

Reagents:

•0.02N H₂SO₄:3 ml. Conc. H₂SO₄ in 1 L distilled water to get 0.01 N H₂SO₄. Take 100 ml of 0.01 N H₂SO₄ + 500 ml distilled water

•Phenolphthalein Indicator: Dissolve 0.5 gm of Phenolphthalein Indicator in 500 ml 95% of ethyl alcohol. Add 500 mL distilled water.

•Methyl orange Indicator: Dissolve 0.5 g of Methyl orange Indicator & dilute to 1000 ml with distilled water.

Procedure:

A. Phenolphthalein Alkalinity:

1. Take suitable volume of sample (25, 50 or 100 ml) in a conical flask.
2. Add 2-3 drops of phenolphthalein indicator.
3. If the sample turns pink in colour, then titrate with 0.02 NH₂SO₄, till the pink colour disappears. Note the volume of acid required as (A).
4. If the pink colour does not exist it indicates that, phenolphthalein Alkalinity is absent. Then continue the titration with Methyl Orange indicator

B. Methyl Orange Alkalinity:

1. Add 2-3 drops of methyl orange to the same flask; continue titration till colour becomes orange. Note down volume of H₂SO₄ consumed as 'B'.
2. If there is no pink colour after adding phenolphthalein, continue according to step 3.
3. Calculate alkalinity in mg/l

Alkalinity Relationship (P and T)

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

Results of Titration	Hydroxide Alkalinity/Caustic Alkalinity as CaCO ₃	Carbonate Alkalinity as CaCO ₃	Bicarbonate Alkalinity as CaCO ₃
P=0	P=0	P=0	M
P= ½ M	0	2P	0
P= M	P= M	0	0
P<1/2 M	0	0	M-2P
P>1/2 M	2P-M	2M-2P	0

Where: P: Phenolphthalein Alkalinity, T: Total Alkalinity

Result:

Sample Used			
Phenolphthalein Alkalinity (mg/L)			
Methyl Orange Alkalinity (mg/L)			
Total Alkalinity (mg/L)			
Hydroxide Alkalinity			
Carbonate Alkalinity			
Bicarbonate Alkalinity			

Inference:

High alkalinity waters may also have a distinctly flat, unpleasant taste. Based on the testing, it is found that the alkalinity of the sample is mg/L. As per the provisional code, alkalinity should not exceed 200 mg/L for potable water. For the fresh water alkalinity ranges between 20 – 100 mg/L. Alkalinity of tested sample is **within/exceeding** the limits specified in the standards. Hence the water sample is **fit /unfit** for drinking.

Ex.No:4

Date: **Determination of Chlorides**

Aim:

To find the amount of chlorides present in the given water sample.

Apparatus Required:

Burette, Conical flask, Pipette, Measuring jar.

Principle:

Silver Nitrate react with chloride to form very slightly soluble white precipitate of AgCl. At the end point when all the chlorides get precipitated free silver ions react with chromate to form reddish brown colour.

Reagents:

1. Silver Nitrate,0.02N
2. Potassium Chromate

Procedure:

1. Take 25 ml water sample in a conical flask and add 1-2 ml Potassium chromate solution.
2. Titrate the contents against 0.02N silver nitrate until a persistent red tinge appears.

Result:

The amount of chloride present in the given water sample is -----

Inference:

The high concentrations of chloride ions mostly results in an unpleasant salty taste of water and it also inhibits the corrosion of plumbing system. Very high chloride content of water may also produce laxative effect. An upper limit of 250 mg/L has been set for the chloride ions.

Tabulation:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of Silver Nitrate (ml)
		Initial Reading	Final Reading		

Calculation:

$$\text{Chloride, mg/l} = \frac{\text{ml of AgNO}_3 \times \text{Normality of AgNO}_3 \times 1000 \times \text{Eq.wt of chlorine}}{\text{Volume of Sample}}$$

Ex.No:5A

Date: **Determination of Phosphates**

Aim

To determine the amount of phosphate present in the given wastewater sample.

Principle

In acidic conditions orthophosphate reacts with ammonium molybdate forming Molybdophosphoric acid, reduced further to molybdenum blue by stannous chloride. The intensity of the blue colour is directly proportional to the concentration of phosphate. The absorbance is noted at 690nm using spectrophotometer.

Apparatus required:

1. Spectrophotometer
2. Lab Glassware
3. Hot Plate
4. Nessler's Tube.

Reagents:

Ammonium molybdate reagent:

25g ammonium molybdate is dissolved in 175ml distilled water. 280ml concentrated sulphuric acid is added to 400ml distilled water and cooled. Molybdate solution is added and the mixture diluted to 1000ml.

Stannous chloride reagent:

2.5g fresh stannous chloride is dissolved in 100ml glycerol, heated on water bath and stirred with the glass rod to hasten dissolution.

Standard phosphate solution:

219.5 mg of dried AR potassium hydrogen phosphate is dissolved in distilled water and made up to 1000ml, where 1ml = 50.0 μ g. of phosphate. 10ml of the stock solution is made up to 1000ml to give 1ml = 0.05 mg. Standards of strength ranging from 0 (blank) to 0.05mg/L at intervals of 0.01mg is prepared by diluting the stock with distilled water

Observation:

SL.NO	CONCENTRATION	ABSORBANCE

Calculation:

$$\text{Phosphates (mg/L)} = \frac{\text{Absorbance of sample} \times \text{Conc.of Std} \times 1000}{\text{Absorbance of Std} \times \text{Sample taken}}$$

Procedure:

1. To 50ml of the filtered sample, 4ml of ammonium molybdate reagent and about 4-5 drops of stannous chloride reagent is added.
2. After about 10 min but before 12 min, the colour developed is measured photometrically at 690nm and calibration curve is prepared.
3. A reagent blank is always run with same treatment with distilled water as sample.
4. The value of phosphate is obtained by comparing absorbance of sample with the standard curve and expressed as mg/L.

Environmental Significance

Presence of phosphates in water and wastewater analysis has a great significance. Phosphate in small concentration are used in water supplies to reduce scale formation, to increase carrying capacity of mains, to avoid corrosion in water mains, to remove iron and manganese in micro quantities and in coagulation especially in acid conditions.

The presence of phosphate in large quantities in fresh waters indicates pollution through sewage and industrial wastes. It promotes growth of nuisance causing micro-organisms. Though phosphate possesses problems in surface waters, its presence is necessary for biological degradation of wastewaters. Phosphorus is an essential nutrient for the growth of organisms and helps for the primary productivity of a body of water.

Result

The amount of Phosphate present in the given sample is ----- mg/l.

Inference:

Any specific permissible limit for phosphates is not available till date since it is a limiting nutrient. However, if the water is to be stored for a longer duration, it is good to have a phosphate content below 0.5 ppm. The water with phosphate content above 0.5 ppm can lead to eutrophication when stored for a longer duration.

Ex.No:5B**Date:** **Determination of Sulphates****Aim:**
To find the amount of Sulphates present in the given water sample.**Apparatus Required:**

Conical Flask, Watt-man filter paper (No 42), crucibles, Measuring Jar, Beaker.

Principle:

Sulphate is precipitated as Barium sulphate in the hydrochloric acid medium by the addition of Barium Chloride solution. The reaction is carried out near the boiling temperature. The precipitate is filtered and then washed to remove the chlorides and ignited and weighed as Barium Sulphate.

Many substances interfere in performing this test. Suspended matter, silica, Nitrate and sulphate lead to the positive errors where the results are on the higher side. Alkali metal sulphates causes the low results. Presence of other metals such as iron and chromium also yield low results due to the formation of metal sulphate.

Reagents:

1. Methyl orange Indicator
2. Barium chloride solution
3. Concentrated Hydrochloric acid.

Procedure:

1. Take 100 ml of sample in a conical flask. Add 2 drops of Methyl orange Indicator to the sample. Adjust pH between 4.5 – 5 by adding few drops of conc Hcl until the colour changes to pink.

2. Add 1 ml of Conc.Hcl heat at 40⁰ C until vapour appears and add Barium chloride Solution until the precipitate forms. This precipitate is heated at 90⁰ C allowing it to boil for 1 hour. Filter it, wash the precipitate with distilled water and filter it with dry ash less filter paper and ignite it in a crucible at 500- 800⁰ C for 1 hour.

3. Cool it in desiccator and weigh the precipitate of Barium sulphate.

Result:

The amount of Sulphates present in the given water sample is -----

Inference:

The presence of sulfate in drinking-water can also result in a noticeable taste; the lowest taste threshold concentration for sulfate is approximately 250 mg/litre as the sodium salt. Sulfate may also contribute to the corrosion of distribution systems.

Tabulation:

SI.No	Description	Weight of crucible		Sulphates
		Empty weight (g)	Empty weight (g) + Filtrate	

Calculation:

$$\text{Amount of Sulphate} = \frac{\text{Mg. of Sulphate} \times 411.5}{\text{ml of Sample}}$$

Ex.No: 6

Date: **Determination of Optimum Coagulant dosage**

Aim:

To find out the optimum coagulant required to precipitate turbid particles present in the water.

Apparatus Required:

Jar test apparatus, Beakers, Turbid water, alum, pipettes .

Principle:

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

Reagents:

Alum solution

Procedure:

1. 200 ml of water sample is taken in each jar. Increasing dose of alum solution is added to each jar i.e., 2,4 and 6...ml and stirred at maximum speed for 1 min. Then stirred slowly for 15 min and allowed to stand for 15 min.
2. The jars are observed and the settling of sediments is noted. The quantity of alum added to the jar containing the clearest solution is noted.
3. Take the sample out of the beaker and test for turbidity in each trial. Plot the curve on x and y axis of the graph sheet. Take the alum dosage in ml along x-axis and turbidity along y-axis.

Result:

The optimum dosage of coagulant required to remove turbidity in the given water sample is _____ml of alum.

Tabulation:

Sample detail/Jar No	Dosage of Coagulant (ml)	Residual Turbidity in NTU

Ex.No:7A

Date: **Determination of Residual chlorine in bleaching**

powder Aim:

To find the amount of residual chlorine present in the given water sample.

Apparatus Required:

Burette, Conical flask, Pipette, Measuring jar.

Principle:

Chlorine is primarily added to the water for destroying the harmful microorganisms. Presence of excess chlorine intensifies the taste and odour of any other compounds in combination with ammonia.

Chlorine is a strong oxidizing agent and liberates Iodine from Potassium Iodide. The liberated Iodine is equivalent to the amount of chlorine and can be titrated against Sodium thiosulphate using starch as an indicator.

Reagents:

1. Acetic Acid
2. Potassium Iodide
3. Sodium Thiosulphate, 0.025N.

Procedure:

1. Take 100ml water sample in a conical flask and add 5ml acetic acid. The pH after addition of acetic acid should be between 3 and 4.

2. Add approximately 1 gm of KI crystals and mix thoroughly with a stirring rod for about 15 minutes keeping it away from the direct sunlight.

3. Add a few drops of starch indicator and titrate against 0.025N sodium thiosulphate until the contents turn colourless from blue.

Result:

The amount of residual chlorine present in the given water sample is -----

Inference:

Residual chlorine is the low level amount of chlorine remaining in the water after a certain period or contact time after its initial application. It constitutes an important safeguard against the risk of subsequent microbial contamination after treatment—a unique and significant benefit for public health.

Tabulation:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of Sodium Thiosulphate (ml)
		Initial Reading	Final Reading		

Calculation:

Residual chlorine ,mg/l = ml of titrate X Normality of Titrate X 1000 X Eq.wt of chlorine

Volume of Sample

Ex.No:7B**Date:** **Determination of Available chlorine in bleaching powder****Aim:**

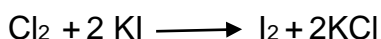
To determine the available chlorine in the given sample of bleaching powder.

Apparatus Required:

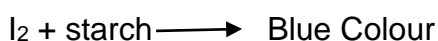
Conical flask, Burette, Pipettes .

Principle:

Chlorine is a strong oxidizing agent and liberates Iodine from Iodide ions.



Starch gives blue colour with Iodine i.e.,



The liberated Iodine is titrated against standard Sodium Thiosulphate-reducing agent



The disappearance of blue colour indicates the completion of reaction with free iodine converted back to iodide.

Reagents:

1. Conc.Acetic Acid
2. Potassium Iodide Crystals
3. Sodium Thiosulphate, 0.025N
4. Starch Indicator

Procedure:

1. 1 gm of fresh bleaching powder is taken and is added to a small quantity of water and made into a fine paste. Some more water is added, stirred well and allowed to settle for a few minutes. It is diluted with distilled water to make upto 1 L and the container is stoppered.

2. 25 ml of the bleaching powder solution is taken in a conical flask and a pinch of Potassium Iodide is added.

3. 2 ml of acetic acid is added and is allowed for the reaction to complete.

4. 1 ml of starch solution is added and the titration is continued till the disappearance of blue colour.

Tabulation:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of Sodium Thiosulphate (ml)
		Initial Reading	Final Reading		

Calculation:

$$\text{Concentration of chlorine, mg/l} = \frac{(V_1 - V_2) \times \text{Normality of titrant} \times 1000 \times \text{Eq.wt of chlorine}}{\text{Volume of Sample}}$$

$$\text{Percentage of chlorine} = \frac{\text{Concentration of chlorine} \times 100}{\dots}$$

Result:

1. The available chlorine in the given sample of bleaching powder is = -----mg/l
2. Percentage of chlorine content in bleaching powder is = -----

Inference:

If the bleaching powder consisted wholly of $\text{Ca}(\text{OCl})\text{Cl}$ it contains 55% of available chlorine—that is, chlorine which can be liberated by treatment with dilute acid; but the commercial variety generally contains other substances, and does not yield more than 36% available chlorine.

Ex.No: 8

Date: Determination of suspended, settleable, volatile and fixed solids

Aim:

To determine the total solids, dissolved solids and settleable solids

Apparatus and equipment:

- a. Electrically heated temperature controlled oven
- b. Weighing balance
- c. Evaporating dish
- d. Pipettes
- e. Measuring cylinder (100mL)
- f. Muffle furnace
- g. Whatmann filter paper (No. 42)

Introduction:

The term 'solid' refers to the matter either filtrable or non-filtrable that remains as residue upon evaporation and subsequent drying at a defined temperature. Further categorisation depends upon depends upon the temperature employed for drying and ignition. Different forms of solids are defined on the basis of method applied for their determination. Solids may affect water or effluent quality adversely in number of ways. Water with high dissolved solids may include an unfavourable physiological reaction in the transient consumer and generally are of inferior palatability. Highly mineralized waters are unsuitable for many industrial applications. High suspended solids in waters may be aesthetically unsatisfactory for such purposes as bathing. Analysis of total solids are important to decide upon the various unit operations and processes in physical and biological wastewater treatment and to assess its performance evaluation. For assessing compliance with regulatory agency, wastewater effluent limitations for various forms of solids act as indicating parameters.

A. Total solids

Principle

Residue left after the evaporation and subsequent drying in oven at specific temperature 103-105°C of a known volume of sample are total solids. Total solids include "Total suspended solids" (TSS) and "Total dissolved solids" (TDS). Whereas loss in weight on ignition of the same sample at 500°C, in which organic matter is converted to CO₂ volatilisation of inorganic matter as much as consistent with complete oxidation of organic matter, are volatile solids.

Sample collection, preservation and storage

The water samples may be collected in resistant glass or plastic bottle. Water has considerable solvent property. There is possibility of increase in mineral content of sample, if water is collected and stored in non-resistant glass bottle. The effect is

pronounced with alkaline water. Exclude particles such as leaves, sticks, fish and lump of fecal matter in the sample. Begin analysis as soon as possible due to impracticality of preservation of sample.

Procedure

1. Take the empty weight of the thoroughly cleaned dish. (W_1)
2. Take 20 ml of a well-mixed sample in the same dish.
3. Evaporate the sample to dryness at 103-105°C in hot air oven. (4-5h)
4. Cool in desiccator, weigh and record the reading (W_2)
5. Take the same crucible and ignite the dish for 30 minutes in a muffle furnace maintained at 550°C.
6. Cool the dish in a desiccator and record final weight (W_3).
7. The concentration is to be calculated in percent by weight.

Procedure for dissolved solids

1. Take the empty weight of the dish (W_4)
2. Take 25 ml of sample and filter it using whatmann filter paper (No. 42) using the filtration assembly.
3. Pour the filtrate into the dish and . Evaporate the sample to dryness at 103-105°C in hot air oven. (4-5 h).
4. Cool in desiccator, weigh and record the reading (W_5)

Procedure for settleable solids

1. Fill an Imhoff cone to the H mark with a well-mixed sample.
2. Settle for 45-min, gently agitate sample near the sides of the cone with a rod or by spinning, settle 15 min longer, and record volume of settleable solids in the cone as ml/L.
3. If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from volume of settled solids.
4. The practical lower limit of measurement depends on sample composition and generally is in the range of 0.1 to 1.0 ml/L. where a separation of settleable and floating materials occurs, do not estimate the floating material as settleable matter.
5. Replicates usually are not required.

Observation:

S.No	Details	Notations	Weight(g)
1	Weight of empty crucible	W1	
2	Weight of crucible with water sample after oven drying	W2	
3	Weight of crucible with water sample after taking it from muffle furnace.	W3	
4	Weight of empty crucible	W4	
5	Weight of crucible with filtrate after oven drying	W5	

Calculation:

Total solids (**TS**) = $(w_2 - w_1) / \text{volume of sample}$

Total dissolved solids (**TDS**) = $(w_5 - w_4) / \text{volume of sample}$

Total suspended solids (**TSS**) = **TS - TDS**

Total fixed solids (**TFS**) = $(w_3 - w_1) / \text{volume of sample}$

Total volatile solids = (**TVS**) = **TS - TFS** =

Total Settleable solids =

Results:

The solids present in the given wastewater are reported below

1. Total solids =
2. Dissolved Solids =
3. Suspended solids =
4. Fixed solids =
5. Volatile solids =
6. Settleable solids =

Inference:

Water can be classified by the amount of TDS per litre:

- Fresh water < 1500 mg/l TDS
- Brackish water = 1500 to 5000 mg/l TDS
- Saline water > 5000 mg/l TDS

Ex.No:9**Date:** Determination Dissolved Oxygen and BOD for the given sample**Aim:**

To determine the BOD in the given wastewater sample.

Apparatus Required:

BOD incubator BOD bottle (300ml)

Principle:

If sufficient oxygen is available in wastewater, the useful aerobic bacteria will flourish and cause the aerobic biological decomposition of wastewater which will continue until oxidation is completed.

The amount of oxygen consumed in this process is the BOD. Polluted waters will continue to absorb oxygen for many months, and it is not practically feasible to determine this ultimate oxygen demand.

Chemicals Required:

- 1.Sodium Hydroxide
- 2.Manganous Sulphate
- 3.Potassium iodide
- 4.Sodium thiosulphate
- 5.Conc.H₂SO₄
- 6.Starch

Reagent Preparation:**1.Manganous Sulphate:**

12 g of Manganous Sulphate is dissolved in 25ml of distilled water.

2.Alkali-Iodide Azide Solution

9 g of Sodium Hydroxide and 2.5 g of Potassium iodide are dissolved in 25ml of distilled water.

3.Sodium thiosulphate Solution (0.025 N)

6.2 g of Sodium thiosulphate is dissolved in 1 litre of water.

4.Starch Solution

Take 1 gm of starch. Prepare paste with distilled water. Make 100 ml with water and boil by stirring and cool it.

Procedure:

1. Distilled water is aerated for 4 hours to attain saturated Dissolved Oxygen (DO) level. In distilled water 1 ml of each nutrients (Phosphate buffer, Magnesium Sulphate, Calcium Chloride and Ferric Chloride) and 1 ml of pre-acclimatized seed per 1 litre of distilled water is added.

2. Two BOD bottles are taken. The wastewater sample 1 ml is taken in the BOD bottles and the aerated water is filled. DO test is conducted for the one BOD bottle sample by

CALCULATION

DO Calculation

Sodium thiosulphate Vs given sample

S.No	Volume of given sample (ml)	Burette reading (ml)		Indicator	End point
		Initial	Final		

Calculation:

$$\text{DO in (mg/l)} = (V_2 \times N \times 8 \times 1000) / V_1$$

V1 = Volume of water sample in ml.

V2 = Volume of Sodium thiosulphate consumed in ml.

N = Normality of Sodium thiosulphate

Initial DO

Final DO

BOD Calculation

$$\text{BOD}_5 \text{ (mg/L)} = [(\text{Initial DO} - \text{Final DO}) \times \text{dilution factor}]$$

Where

$$\text{Dilution factor} = \frac{\text{Volume of diluted sample}}{\text{Volume of wastewater sample added}}$$

the following steps and initial DO is noted.

3. Distilled water is aerated for 4 hours to attain saturated Dissolved Oxygen (DO) level. In distilled water 1 ml of each nutrients (Phosphate buffer, Magnesium Sulphate, Calcium Chloride and Ferric Chloride) and 1 ml of pre-acclimatized seed per 1 litre of distilled water is added.

4. Two BOD bottles are taken. The wastewater sample 1 ml is taken in the BOD bottles and the aerated water is filled. DO test is conducted for the one BOD bottle sample by the following steps and initial DO is noted

5. 1 ml of Manganese Sulphate solution is added, followed by 1 ml of Alkali Iodide Azide reagent. Then the bottle is mixed twice and allowed to precipitate settle.

6. 1 ml of Sulphuric Acid is added and mixed twice. 200 ml of sample is taken and titrated against Sodium thiosulphate solution with starch indicator.

7. Disappearance of blue colour is taken as end point. Volume of Sodium thiosulphate consumed is noted.

8. Another bottle is placed in incubator at 20° C. After 5 days DO test is conducted and final DO is noted.

Result:

BOD₅ of given sample at 20°C = mg/l

Inference:

BIS Standard of wastewater effluent BOD to be discharged on land for irrigation purpose is 500 mg/l. The amount of BOD obtained at 20°C is . So, this wastewater can/cannot be used for irrigation purpose.

Ex.No: 10

Date: **Determination of COD for given sample**

Aim:

To determine the chemical oxygen demand (COD) exerted by the given waste water sample.

Apparatus Required:

Reflux Apparatus, Burette, Hot plate/Heating mantle

Principle:

The organic matter present in sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 and H_2O . The excess of $K_2Cr_2O_7$ remaining after the reaction is titrated with $Fe(NH_4)_2(SO_4)_2$. The dichromate consumed gives the oxygen required for the oxidation of organic matter.

Reagents:

1. Standard Potassium Dichromate 0.2N
2. Sulphuric Acid with reagent ($Conc. AgSO_4 + H_2SO_4$)
3. Std. Ferrous Ammonium Sulphate (0.1N)
4. Ferroin Indicator
5. Mercuric Sulphate
6. Silver Sulphate

Procedure:

1. Take 10 ml of sample.
2. 5 ml of more Conc Dichromate solution are placed in a flask together with glass beads.
3. Add slowly 15 ml of H_2SO_4 containing $AgSO_4$ and mix thoroughly.
4. Add pinch of mercurous sulphate ($HgSO_4$) and silver sulphate (Ag_2SO_4).
5. Connect the flask to condenser. Mix the contents thoroughly before heating. Improper mixing may result in bumping and the sample may be blown out.
6. Reflux for a minimum period of 2 hours. Cool and wash down the condenser with distilled water.
7. Dilute the sample to make up to 40 ml with distilled and cool.

Tabulation:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of FAS (ml)
		Initial Reading	Final Reading		

Calculation:

Quantity of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ added for blank (A) =

Quantity of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ added for sample (B) =

$$\text{Chemical Oxygen Demand (COD)} = \frac{(\text{A}-\text{B}) \times \text{N} \times 8 \times 1000 \times \text{Dilution factor}}{\text{Volume of Sample}}$$

8. Add 2-3 drops of Ferroin indicator. Mix thoroughly and titrate it against 0.1N $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$. Sharp colour changes from blue-green to wine red indicate the end point.

9. Reflux the blank solution in the same manner using distilled water instead of sample.

Result:

The COD for the given water sample is -----

Inference:

The BIS standard for COD of wastewater effluent to be discharged into marine coast is 250 mg/l. The amount of COD obtained in the given sample is . So, this water can/cannot be disposed in the marine coast.

Ex.No: 11A

Date:

Determination of Sodium for given sample

Aim:

To determine the amount of sodium present in the given wastewater sample.

Apparatus required:

- 1.Flame Photometer,
- 2.Lab Glassware
- 3.Wattman Filter Paper.

Reagents required:

- 1.Deionised water
- 2.Stock sodium solution: 2.542g of Sodium chloride of dried (at 140o C) was dissolved in 1000ml distilled water to give 1ml = 1mg of sodium.
- 3.Working Potassium solution:

Working standards of suitable strengths were prepared from the stock solution.

Procedure:

- 1.The filter of the flame photometer is set to 589nm (marked for Sodium, Na).
- 2.By feeding distilled water the scale is set to zero and maximum using the standard of highest value.
- 3.A standard curve between concentration and emission is prepared by feeding the standard solutions.
- 4.The sample is filtered through filter paper and fed into the flame photometer and the concentration is found from graph or by direct readings.

Result:

The amount of Sodium present in the given sample is----- mg/l.

Observation:

Sl.No	Concentration	Absorbance

Ex.No: 11B

Date: **Determination of Potassium for given sample**

Aim

To determine the amount of potassium present in the given wastewater sample.

Principle:

Trace amount of potassium can be determined by direct reading of flame photometer at a specific wavelength of 766.5nm by spraying the sample into the flame. The desired spectral lines are then isolated by the use of interference filters or suitable slit arrangements. The intensity of light is measured by the phototube.

Working principle of Flame photometer:

The emission of characteristic radiations by alkali and alkaline earth metals and the correlation of the emission intensity with the concentration of the element form the basis of flame photometry. The principle of the flame photometer depends on the "Emission Spectroscopy" in which the electrons of the metals after absorbing energy get excited from ground state to higher energy level and return back to the ground state with emission of light. The sample under test is introduced into flame in solution by means of atomizer. The radiation from the flame enters a dispersing device and isolates it (radiation) from the flame to the desired region of the spectrum. The phototube measures the intensity of isolated radiation, which is proportional to the concentration of the element present in the sample.

Apparatus required:

- 1.Flame Photometer,
- 2.Lab Glassware
- 3.Whatman Filter Paper.

Reagents required

- 1.Deionised distilled water.
- 2.Stock potassium solution:1.907g of dried Potassium chloride, is dissolved in 1000ml of distilled water, to give 1ml = 1mg of potassium
- 3.Working Potassium solution:

Working standards of suitable strengths are prepared from the stock solution.

Procedure:

- 1.The filter of the flame photometer is set at 766.5nm (marked for Potassium, K) the flame is adjusted for blue colour.
- 2.The scale is set to zero and maximum using the highest standard value.
- 3.A standard curve of different concentration is prepared by feeding the standard solutions. The sample is filtered through the filter paper and fed into the flame photometer.
- 4.The concentration is found from the standard curve or as direct reading.

Result:

The amount of Potassium present in the given sample is----- mg/l.

Observation:

Sl.No	Concentration	Absorbance

Ex.No: 12

Date: Determination of SVI of Biological sludge and microscopic examination

Aim:

To determine SVI of Biological sludge

Sludge Volume Index (SVI)

Sludge volume index is a common measurement that we use to determine sludge quality. In wastewater treatment processes, a good quality sludge will generally produce a good quality effluent. SVI takes into consideration the mixed liquor suspended solids concentration (MLSS) and the volume that a certain amount of sludge takes up. Generally, a good quality sludge has an SVI in the range of 50-150 mL/gram.

Apparatus Required:

Measuring Cylinder, Distilled water, Whatman filter paper, Funnel, Hot air oven., Weighing Balance

Procedure:

(A) Measurement of Sludge Volume Index

In order to calculate sludge volume index (SVI), you will need two numbers. The first number comes from a 30-minute settleability test, where 1 liter of the mixed liquor sample from the aeration tank is poured into a container called a settleometer. The sludge is allowed to settle for 30 minutes, and the volume of the settled sludge is measured in mL/L.

(B) Measurement of MLSS

The other number used in the sludge volume index (SVI) calculation comes from a MLSS test. It simply determines the suspended solids concentration of the sample from the aeration tank, in mg/L.

1. Take a Whatman filter paper and make it as a cone shaped. Keep it in oven for drying. Then measure the initial weight of the filter paper(W_i).
2. Take a funnel and keep it on the measuring cylinder. Then place the dried filter paper in the funnel. Now pour the 25 ml sample in the filter paper. Allow it to settle for 10-15 min.
3. Keep the filter paper with residue in the hot air oven for 2 hrs @ 100°C. After cooling measure the final weight of the filter paper(W_f).

Sludge volume index (SVI) is calculated by dividing the settleability by the MLSS concentration. The SVI is always expressed in mL/g. Finally the microscopic examination is to be done for SVI.

Calculation:

$$\text{SVI (mL/g)} = \frac{\text{Settled sludge volume mL/L} \times 1000 \text{mg/g}}{\text{MLSS mg/L}}$$

$$\text{MLSS (g/L)} = \frac{(W_f - W_i)}{\text{Volume of sample}}$$

$$\text{MLSS (mg/L)} = \frac{(W_f - W_i) \times 10^6}{\text{Volume of sample}}$$

Results:

The SVI of given Biological sludge is _____.

Interpreting Results:

The higher the SVI, the slower the sludge settles. If the sludge is settling too slow, this can result in loss of suspended solids over the clarifier weirs. If the SVI settles too quickly, and the SVI is <50 this can be due to pin floc, which is commonly (but not always) caused by old sludge. It is recommended to perform the SVI test every day. It is beneficial to determine the SVI for 30 minute as well as occasionally for 60 minute settling. The majority of the settling should occur within the first 30 minutes. If there is a change in your SVI, it is recommended to perform a microscopic evaluation to determine the cause. It is also useful to leave the settled sludge in the container and observe if there is a rise time, in which the sludge floats. This indicates denitrification. If the rise time is <2 hours, there is risk of floating sludge in the clarifier and steps need to be taken to reduce nitrate, BOD, and increase oxygen going into the clarifier.

Ex.No: 13

Date: Determination of MPN index of given water sample

Aim:

To determine the MPN index of given water sample

Principle:

Water to be tested is diluted serially and inoculated in lactose broth, coliforms if present in water utilize the lactose present in the medium to produce acid and gas. The presence of acid is indicated by color change of the medium and the presence of gas is detected as gas bubbles collected in the inverted durham tube present in the medium. The number of total coliforms is determined by counting the number of tubes giving positive reaction (*i.e both color change and gas production*) and comparing the pattern of positive results (*the number of tubes showing growth at each dilution*) with standard statistical tables.

MPN test is performed in 3 steps

1. Presumptive test
2. Confirmatory test
3. Completed test

1. Presumptive Test:

It looks for the presence of fecal coliforms in the water sample.

Requirements:

- Lactose broth (both single and double strength lactose broth)
- Cotton plug
- Sterile pipette
- Durham tubes-sterilize by autoclaving at 15lbs pressure(121 degree celcius) for 15 minutes.
- Test tubes of various capacities(20ml,50ml)

Procedure:

1. Prepare the lactose broth medium in both single and double strength concentration.
2. Take 3 sets of test tube, each consists of 5 numbers of test tubes.
3. Fill 10 ml of the double strength lactose broth in 1 set (5 no.s) of test tubes of capacity of 50 ml and add a durham tube in inverted position.
4. Check for no formation of bubbles.

5. Fill 10 ml of the single strength lactose broth in 2 sets (10 no.s) of test tubes of capacity of 20 ml and durham tube in inverted position.
6. Check for no formation of bubbles
7. Sterlize the 3sets of test tubes with lactose broth by autoclaving.
8. Then the first set is inoculated with 10 ml of sample in each test tube.
9. Second set is inoculated with 1ml of sample in each test tube.
10. Third set is inoculated with 0.1ml of sample in each test tube.
11. Then incubated all the tubes at 37 degree Celsius for 24 hrs. If no tube appears positive re-incubate up to 48 hrs.
12. Tubes then shows presence of gas are scored + and those with no gas as–
13. Compare the number of tubes giving positive reaction to a standard chart and record the number of bacteria present per 100 ml of sample.

2. Confirmed Test:

It looks for the confirmation of bacteria present in the sample. The tubes that show positive in the presumptive test may contain E.coli. This is confirmed by inoculating it in Eosin Methylene Blue (EMB) agar medium. It is an nutrient supplier for the growth of bacteria and it kills the gram positive bacteria present in the sample unwantedly and enhances the growth of E.coli.

Requirements:

- Medium: Eosin methylene blue (EMB).
- Petri plates
- Loop

Procedure:

1. Sterlize the EMB agar medium and the petri plates by autoclaving.
2. Then pour the EMB agar medium in the petri plates and leave it until it gets solidified.
3. Take a loop of a culture from the first set test tube and make a streak in the petri plate with the medium and allow it to grow.
4. The growth of bacteria in the EMB agar medium with glossy green shade will confirm the presence of E.coli.

3. Completed Test:

Requirements:

- Nutrient agar slant
- Test tubes
- Petri plates
- Lactose broth
- Loop
- Durham tube

Procedure:

1. The organisms which grew on the confirmed test media are inoculated on to a nutrient agar slant and a tube of lactose broth.
2. After 24 hours at 37 degree celcius, the lactose broth is checked for the production of gas, and a Gram stain is made from organisms on the nutrient agar slant.
3. If the organisms is a Gram-negative, non-spore forming rod and produces gas in the lactose tube, then it is positive that coliforms are present in the water sample.

Advantages of MPN :

- Ease of interpretation, either by observation or gas emission
- Sample toxins are diluted
- Effective method of analyzing highly turbid samples such as sediments, sludge, mud, etc.
- That cannot be analysed by membrane filtration.

Disadvantages of MPN:

- It takes a long time to get the results
- Results are not very accurate
- Requires more hardware (glassware) and media
- Probability of false positives

Ex.No:

Date:

Determination of Oil and Grease

Aim :

To find out the amount of oil and grease matters present in given waste water sample

Necessity for this experiment:

- The oil and grease contents of domestic and certain industrial wastes and the sludge, is of an important consideration in the handling and treatment of these material for ultimate disposal.
- Knowledge of the quality of the oil and grease present is helpful in proper design and operation of wastewater treatment system.

Background:

- FOG is defined as a compound either in liquid or solid state, basically from animal and vegetable sources. Oil is defined as tri-glycerides that are liquid at room temperature. Grease is a general classification for fats, oil, waxes and soaps that have a negative effect on the wastewater treatment system.
- Oil and grease are insoluble in water and basically made of esters of glycerol, fatty acids or triglycerides. One of the most significant characteristics of natural oil is low solubility in water with high tendency in dissolving in organic solvents.
- The specific gravity of these compounds is less than that of water and they easily become saponified in alkaline media.

Apparatus Required:

- Separating funnel
- Water bath
- Filter paper

Reagents:

- H₂SO₄
- Petroleum ether

Procedure:

- Take a 200 – 250 ml of sample in separating funnel.
- Add 10 ml of H₂SO₄ .(1:2) and 50 ml of petroleum ether to the sample.
- Shake well and if suspension prevails, add small amount of ethyl alcohol. Keep for some time for separation of two layers. The upper one is petroleum ether with oil and grease and the lower one is the sample.
- Discard lower layer of the sample through the separating funnel.
- Take a pre-weighed dish or a small beaker and collect the petroleum ether from the separating funnel through a filter paper.
- Evaporate the petroleum ether on a water bath and take the final weight of the dish or beaker after cooling in dessicator. Never heat petroleum ether on a flame.

Result:

The amount of Oil & Grease present in the given water sample is mg/l.

Inference:

As per IS 10500 – 2012, the permissible value of oil and grease content in,

- Inland surface water = 10 mg/l
- Public sewer line = 20 mg/l
- Land for irrigation = 10 mg/l

Calculation:

Oil and grease in mg/l = $[(B-A) \times 1000 \times 1000]/V$ Where,
A = Initial weight of dish in 'g' B = Final weight of dish in 'g'
V = volume of sample taken in 'ml'

Ex.No
Aim:

Determination Of Calcium In The Given Wastewater Sample

To determine the amount of calcium present in the given water sample.

Principle:

When EDTA (Ethylene-diamine tetra acetic acid) is added to the water containing calcium and magnesium, it combines first with calcium. Calcium can be determined directly with EDTA when pH is made sufficiently high such that the magnesium is largely precipitated as hydroxyl compound (by adding NaOH and iso-propyl alcohol).

Apparatus required:

1. Burettes
2. Pipette
3. Conical Flask
4. Beaker

Reagents:

1. Ammonia Buffer
2. EBT indicator
3. 0.02N EDTA

Procedure:

1. Pipette out 20 ml of sample water and transfer it to a conical flask.
2. Measure 2 to 3 ml of ammonia buffer solution and add it to the water sample. so that pH will be maintained between 12 to 13.
3. Add 2 to 3 drops of EBT indicator to the water sample.
4. Now the sample turns into pink colour.
5. This colour change is due to Ca and Mg contents present in water.
6. Fill the burette with standard EDTA solution.
7. Titrate the water sample against EDTA solution in the burette till all Mg and Ca ions present in sample reacts with EDTA to form metal EDTA complex by changing the colour of the sample to steel blue that is end point.

Environmental Significance:

The relative amounts of Calcium hardness, Carbonate and non-Carbonate hardness present in water are the factors while determining the most economical type of softening process. Hard water typically contains high concentrations of Ca and Mg cations, which interfere with the use of the water for many applications. These ions diminish the effectiveness of soaps and detergents for cleaning operations. They diminish the drinking of water and they contribute to the accumulation of insoluble salt deposits in storage vessels or plumbing.

Result:

The amount of Calcium present in the given sample is ----- mg/l

Observation

EDTA Vs Wastewater sample

<u>S.No</u>	Volume of given sample (ml)	Burette Reading (ml)		Volume of EDTA consumed (ml)	Hardness (mg/L)
		<u>Initial</u>	<u>Final</u>		

Calculation:

Volume of EDTA = ml
Normality of EDTA = N
Volume of Sample taken = ml
Equivalent weight of CaCO₃ = 50
Molecular weight of Calcium = 40.078
Molecular weight of Calcium Carbonate = 100

$$\text{Hardness as CaCO}_3 \text{ equivalent mg/l} = \frac{\text{Volume of EDTA} \times N \times 50 \times 1000}{\text{Volume of sample taken}}$$

$$\text{Calcium Ca mg/l} = \frac{\text{Ca hardness in mg/l as CaCO}_3 \times \text{molecular weight of Ca}}{\text{molecular weight of Calcium carbonate}}$$

VIVA QUESTIONS

1. State whether the following statement is True or False.
Carbonate hardness can be removed by adding lime to water.

- a) True
- b) False

Answer: a

Explanation: Carbonate hardness is due to the presence of carbonates and bicarbonates of calcium and magnesium in water and can be removed by boiling or by adding lime to it.

2. Which of the following statement is wrong regarding permanent hardness?

- a) It is also called carbonate hardness
- b) It is due to the presence of sulfates, chlorides and nitrates of calcium and magnesium
- c) It cannot be removed by boiling
- d) It requires special methods of water softening to get removed

Answer: a

Explanation: Permanent hardness is called non-carbonate hardness whereas temporary hardness is called carbonate hardness.

3. One degree of hardness is equivalent to _____ ppm

- a) 2
- b) 1
- c) 10
- d) 100

Answer: b

Explanation: Water is said to have one degree of hardness when its soap destroying power is equivalent to the effect of 14.25mg of calcium carbonate in one litre of water.

4.

Degree of hardness	Nature of water
1	Extremely soft water
2	Very soft water
3	Soft water
9	?

What can be filled in place of the question mark in the above table?

- a) Excessive hard water
- b) Reasonably soft water
- c) Very hard water
- d) Hard water

Answer: d

Explanation: When the degree of hardness is 9, then it indicates the hard water. For excessive hard water, it is 15 and for very hard water, it is 11.

5. In which method of determining total hardness of water is based on the premise that hardness producing substance react with soap and form insoluble compounds before lather is produced?

- a) Clark's method
- b) Hehner's method
- c) Versenate method
- d) EDTA method

Answer: a

Explanation: In Clark's method, total hardness is found by determining the standard soap solution required to obtain permanent lather with the water sample of known volume.

6. What is the indicator used in EDTA method?

- a) Potassium chromate
- b) Potassium dichromate
- c) Potassium chloride
- d) Erio chrome, black T

Answer: d

Explanation: Hardness is determined by using Ethylene diamine tetra-acetic acid using Erio chrome, black T as indicator at a pH between 8.5 and 11.

7. The permissible limit of pH preferred for potable water is ____ ppm.

- a) 6.5-9
- b) 7-8.5
- c) 10-14
- d) 0-7

Answer: a

Explanation: The permissible limit of pH for potable water should not exceed 9 and should not be less than 6.5 and the preferred limit is 7-8.5.

8. Given list 1 and list 2, find which of the following pair is correctly matched?

List 1 (Chemical substance)	List 2 (Permissible limit in ppm)
A. Lead	1. 0.05
B. Phenol	2. 0.001
C. Zinc	3. 0.01
D. Chromium	4. 5
	5. 0.2

advertisement

- A-5
- b) B-3
- c) C-4
- d) D-2

Answer: c

Explanation: Lead has a permissible limit of 0.05ppm, phenol has 0.001ppm, zinc has 5ppm and chromium has a permissible limit of 0.05ppm in water.

9. What is the concentration of H⁺ ions in moles/L in water if the pOH value is 6?

- a) 10⁻⁶
- b) 10⁻⁷
- c) 10⁻⁸
- d) 10⁻⁹

Answer: c

Explanation: $\text{pH} + \text{pOH} = 14$

$\text{pOH} = 6$, so $\text{pH} = 14 - 6 = 8$

$\text{H}^+ = 10^{-\text{pH}} = 10^{-8} \text{ moles/L}$.

10. The carbonaceous demand occurs due to oxidation of

- a) Ammonia
- b) Sulfur
- c) Organic matter
- d) Nitrogen

Answer: c

Explanation: The carbonaceous demand occurs due to oxidation of organic matter. The carbonaceous demand is also called as the initial demand.

11. The biochemical oxygen demand is computed by

- a) Dissolved oxygen / Dilution factor
- b) Dissolved oxygen + Dilution factor
- c) Dissolved oxygen – Dilution factor
- d) Dissolved oxygen * Dilution factor

Answer: d

Explanation: $\text{BOD} = \text{Dissolved oxygen} * \text{Dilution factor}$.

Where, Dilution factor = Volume of diluted sewage sample / Volume of undiluted sewage sample.

12. The full form of BOD is

- a) Biodegradable oxygen demand
- b) Biological oxygen demand
- c) Biochemical oxygen demand
- d) Bandwidth on demand

Answer: c

Explanation: The full form of BOD is biochemical oxygen demand. It is measured in ppm or mg/L.

13. _____ is the amount of oxygen required to oxidize only organic matter in sewage.

- a) Turbidity
- b) BOD
- c) COD
- d) DO

Answer: b

Explanation: BOD is the amount of oxygen required to oxidize only organic matter in sewage. It is always less than COD as COD oxidizes both organic and inorganic matter.

14. The design value of the velocity of flow in a flocculator is _____

- a) 0.2-0.8m/s
- b) 0.3-0.5m/s
- c) 0.6-0.8m/s
- d) 0.1-0.5m/s

Answer: a

Explanation: The normal value of the velocity of flow in a flocculator is 0.4m/s and its range is 0.2-0.8m/s.

15. _____ is an operation designed to force agitation in the fluid and induce coagulation.

- a) Sedimentation
- b) Flocculation
- c) Disinfection
- d) Aeration

Answer: b

Explanation: Flocculation is an agitating process in which destabilized particles are brought into contact to promote agglomeration.

16. What is the normal value of the detention period adopted in a flocculator for design purpose?

- a) 30min
- b) 60min
- c) 90min
- d) 100min

Answer: a

Explanation: The detention period in a flocculator for design purpose should be in the range of 10-40min and its normal value should be 30min.

17. Which of the following is termed as free available chlorine?

- a) Hypochlorous acid
- b) Hypochlorite ions
- c) Molecular chlorine
- d) Hypochlorous acid, Hypochlorite ions, Molecular chlorine

Answer: d

Explanation: The chlorine existing in water as hypochlorous acid, hypochlorite ions, molecular chlorine is termed as free available chlorine.

18. The permissible limit of free residual chlorine is

- a) 0.02ppm
- b) 0.2ppm
- c) 1ppm
- d) 2ppm

Answer: b

Explanation: The water is satisfactorily disinfected if the free residual chlorine is 0.2 ppm at the contact period of 10 minutes.

19. What is the chemical formula of bleaching powder?

- a) Ca (OCl)_2
- b) Ca (OCl)
- c) Ca (OCl)_3
- d) CaCl_2

Answer: a

Explanation: The chemical formula of bleaching powder is Ca (OCl)_2 . It is called as chlorinated lime.

20. The permissible limit of turbidity of domestic water is _____ppm.

- a) 5-10
- b) 1-5
- c) 10-50
- d) 10-30

Answer: a

Explanation: According to WHO, the permissible limit for drinking water is 5NTU.

21. What is the full form of NTU in context with turbidity?

- a) Number of transfer unit
- b) Neurological turbidity unit
- c) Nephelometric turbidity unit
- d) Network terminal unit

Answer: c

Explanation: The value of turbidity measured by nephelometer is expressed in terms of NTU. Sometimes, it is referred as FTU or Formazin turbidity unit.

22. The size of suspended solids lies in the range of _____

- a) $10^{-3} - 10^{-6}$ mm
- b) $10^3 - 10^6$ mm
- c) $10^{-1} - 10^{-3}$ mm
- d) $10^1 - 10^3$ mm

Answer: c

Explanation: Size of suspended solids – $10^{-1} - 10^{-3}$ mm, size of colloidal solids – $10^{-3} - 10^{-6}$ mm, size of dissolved solids – $< 10^{-6}$ mm.

23. Suspended solids are measured by which of the following?

- a) Turbidity rod
- b) Gravimetric test
- c) Chromatography
- d) Jackson's turbidity meter

Answer: b

Explanation: Turbidity rod and Jackson's turbidity meter is used to find turbidity whereas chromatography is used to detect odour. Hence b is the correct answer.

24. The maximum permissible limit for suspended solids is _____

- a) 10 mg/l
- b) 20 mg/l
- c) 30 mg/l
- d) 40 mg/l

Answer: c

Explanation: The maximum permissible limit for suspended solids is 30mg/l for testing waste water which is set by the Environmental Protection Agency (EPA).

25. Identify the correct relation between the following?

- a) Dissolved solid = Total solid + Suspended solid
- b) Dissolved solid = Total solid – Suspended solid
- c) Total solid = Dissolved solid / Suspended solid
- d) Dissolved solid = Suspended solid – Total solid

Answer: b

Explanation: Dissolved solid is the difference between total solid and suspended solid. Suspended solid is non-filterable solid whereas dissolved solid is filterable solid.

26. Which method is used to measure the color of water?

- a) Gravimetric analysis
- b) Chromatography
- c) Tintometer method
- d) Hydrometer analysis

Answer: c

Explanation: The colors in water are measured by Tintometer method. It is measured on Burgess scale or cobalt scale by Nessler's tube.

27 TCU (True Color Unit) is equivalent to _____

- a) The color produced by 1 g of platinum cobalt
- b) The color produced by 1 mg of platinum cobalt
- c) The color produced by 1 mg of platinum cobalt in 1L of distilled water
- d) The color produced by 1 mg of platinum cobalt in 1mL of distilled water

Answer: c

Explanation: 1 TCU (True Color Unit) is equivalent to color produced by 1 mg of platinum cobalt in 1L of distilled water. The true color unit is also called as Hazen unit.

28. The range for threshold odour number is

- a) 0-3
- b) 1-5
- c) 1-3
- d) 0-5

Answer: c

Explanation: The maximum value of Threshold odour number is 3 and its range is 1-3. The threshold odour number is used to measure the intensity of taste and odour.

29. ---represents the bacterial density that is most likely to be present in water.

- a) BOD
- b) COD
- c) MPN
- d) Coliform index

Answer: c

Explanation: Most Probable Number is a number that indicates the bacterial density present in water. BOD and COD are the terms related to the oxygen required by microorganism to oxidize organic and inorganic matter.