

Techno India NJR Institute of Technology



Environmental Engineering Design and Lab (6CE4-21)

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Syllabus

3rd Year - VI Semester: B.Tech. (Civil Engineering)

6CE4-21: Environmental Engineering Design and Lab

Credit: 1.5

OL+OT+3P

Max. Marks: 75(IA:45, ETE:30)

End Term Exam: 3 Hours

Design

1. Population forecasting and water demand
2. Water Quality parameters
3. Design of Sedimentation tanks, coagulation and flocculation tanks
4. Design of rapid and slow sand filters
5. Design of disinfection units and transmission systems
6. Design of Sewer lines and storm water systems
7. Design of aerobic and anaerobic treatment units
8. Design of suspended and attached growth systems

Lab.

1. Physical Characterization of water: Turbidity, Electrical Conductivity, pH
2. Analysis of solids content of water: Dissolved, Settleable, suspended, total, volatile, inorganic etc.
3. Alkalinity and acidity, Hardness: total hardness, calcium and magnesium hardness
4. Optimum coagulant dose
5. Chemical Oxygen Demand (COD)
6. Dissolved Oxygen (D.O) and Biochemical Oxygen Demand (BOD)
7. Break point Chlorination
8. Bacteriological quality measurement: MPN,

Course Overview:

Environmental Engineering combines the principles of engineering, chemistry, and biology to provide safe water, sanitation, and clean air. With an increasing demand for safer environments, the need for highly trained environmental engineers is growing. Environmental engineers design systems that protect people and planet from the mismanagement of toxic and hazardous waste. And they develop solutions to rehabilitate impacted terrestrial and aquatic environments. From wastewater treatment systems to air quality management technologies, environmental engineers are creating a cleaner and safer tomorrow

Course Outcomes:

| CO.NO. | Cognitive Level | Course Outcome |
|--------|--------------------|--|
| 1 | Analysis | Understand about the water quality parameters and their permissible limits as per the standards. |
| 2 | Evaluation | Analyze the physical tests to be conducted for the water before supply. |
| 3 | Synthesis | Analyze chemical tests to be conducted for the water before supply. |
| 4 | Synthesis | Accumulate the information about water supply fittings. |
| 5 | Application | Calculate physical chemical properties by lab experiments for sewage sample. |

Prerequisites:

1. Analyze characteristics of water and wastewater
2. Students will develop an appreciation for the importance of environmental engineering as a major factor in preserving and protecting human health and the environment

Course Outcome Mapping with Program Outcome:

| Course Outcome | PO1 | PO2 | PO3 | PO4 | PO5 | PO6 | PO7 | PO8 | PO9 | PO10 | PO11 | PO12 | PSO1 | PSO2 | PSO3 |
|--------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|------|------|------|------|------|------|
| CO363.1 | 3 | 2 | 2 | 2 | 2 | 2 | 2 | 1 | 2 | 1 | 1 | 1 | 2 | 2 | 1 |
| CO363.2 | 3 | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 2 | 1 | 2 | 2 | 2 | 1 | 1 |
| CO363.3 | 3 | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 2 | 1 | 2 | 2 | 2 | 1 | 1 |
| CO363.4 | 2 | 2 | 3 | 3 | 2 | 1 | 3 | 1 | 1 | 1 | 1 | 2 | 2 | 2 | 1 |
| CO363.5 | 2 | 2 | 2 | 3 | 1 | 1 | 2 | 1 | 1 | 2 | 1 | 2 | 2 | 2 | 1 |
| CO363 (AVG) | 2.6 | 2 | 2.2 | 2.4 | 1.8 | 1.6 | 1.8 | 1 | 1.6 | 1.2 | 1.4 | 1.8 | 2 | 1.6 | 1 |

Course Coverage Module Wise:

| | |
|----------------|---|
| Lab No. | Experiments List According to RTU Syllabus |
|----------------|---|

| | |
|---|---|
| 1 | Physical Characterization of water: Turbidity, Electrical Conductivity, pH |
| 2 | Analysis of solids content of water: Dissolved, Settleable, suspended, total, volatile, inorganic etc |
| 3 | Alkalinity and acidity, Hardness: total hardness, calcium and magnesium hardness |
| 4 | Optimum coagulant dose |
| 5 | Chemical Oxygen Demand (COD) |
| 6 | Dissolved Oxygen (D.O) and Biochemical Oxygen Demand (BOD) |
| 7 | Break point Chlorination |
| 8 | Bacteriological quality measurement: MPN |
| 12 | Development of Plan, Front Elevation and Sectional Elevation from line diagram |
| Advance List of Experiment Beyond The RTU Syllabus | |
| 1 | Field Sample Collection of Water and Sewage |

Faculty Lab Manual Link

1. https://r.search.yahoo.com/_ylt=AwrxzALl4qhxC3UAPWu7HAX.;_ylu=Y29sbwNzZzMEcG9zAzEEdnRpZAMEc2VjA3Ny/RV=2/RE=1638749029/RO=10/RU=https%3a%2f%2fwww.iare.ac.in%2fsites%2fdefault%2ffiles%2flab1%2fEnvironmental_Engineering%2520_Laboratory_Lab_MANUAL.pdf/RK=2/RS=wegI0PvdQ_xKJ3fWJJE2IP5K808-

Assessment Methodology

Methodology:

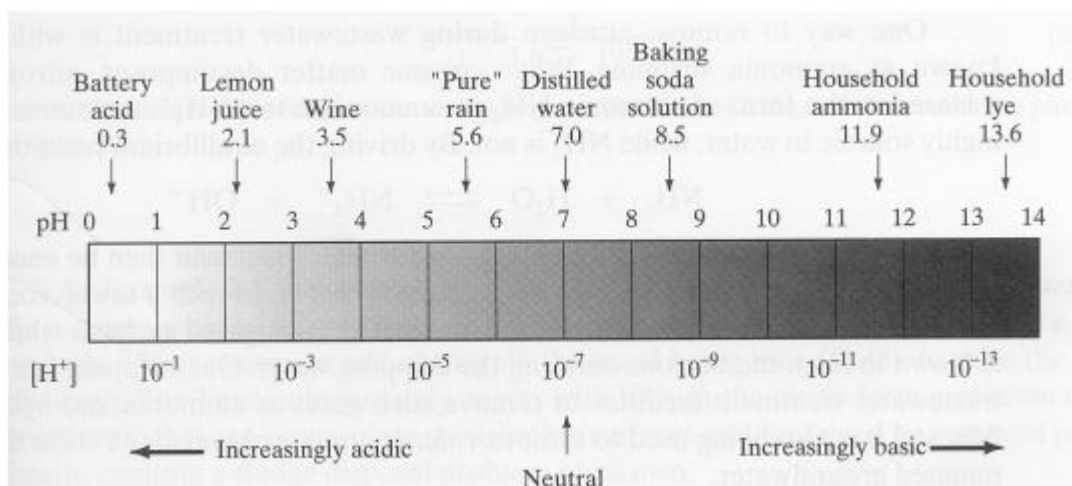
1. Practical exam Of Environmental lab Experiment
2. Internal exams and Viva Conduct.
3. Final Exam (practical paper) at the end of the semester.

pH, Conductivity and Turbidity

Objective: Measure pH, conductivity and turbidity

Background

I) **pH:** The pH is one of the basic water and wastewater characteristics. It expresses the intensity of acid or alkaline conditions by indicating the hydrogen ion activity. Some of the processes in water quality engineering that require pH monitoring and control are the following: disinfection, coagulation, softening, biological treatment etc. Natural waters usually have pH values close to neutral. Figure shows pH values of commonly used household products.



(ii) **Conductivity:** Conductivity, k , is a measure of the ability of an aqueous solution to carry an electric current due to presence of ions, their concentration, mobility and temperature. Selection of Cell constant Conductivity cells of different cell constants are chosen mainly to ensure that the actual resistance between the plates, when the cell is immersed in the solution, is within practical measurable range. Inaccuracy creeps in if the resistance to be measured is too high or too low. A safe practical range is 1 M ohms to 10 ohms. A general guide for selection of cell constant is given in Table

| Cell Constants & Conductivity Range | |
|-------------------------------------|-------------------------------------|
| Cell Constant K | Conductivity Range |
| 0.001 | 0 to 10 $\mu\text{S}/\text{cm}$ |
| 0.1 | 0.1 to 1000 $\mu\text{S}/\text{cm}$ |
| 1 | 10 μS to 10 mS/cm |
| 10 | 100 μS to 1 S/cm |

Selection of Standard KCl solution As a rule of thumb, wherever a measurement is based on comparison with a known standard, the standard chosen should be closer to the value under measurement. In view of the above, Systronics Conductivity Meter Model 306, offers three standard solutions with which the unit can be standardized. The conductivity and temperature co-efficient of the three at / around 25°C are given in Table 2

| Temperature correction for conductivity | | |
|---|--------------------------|-----------------|
| Concentration at ion 25°C | Conductivity at ion 25°C | Temp Correction |
| 0.1 M KCl | 12.88 mS | + 1.90 % |
| 0.01 M KCl | 1.413 mS | + 1.94 % |
| 0.001 M KCl | 0.146 mS | + 2.04 % |

The above table can be used to calculate the conductivity (approx) at any other ambient temperature.

Example: Calculation of conductivity of 0.01 KCl at 30°C
 $C_{30^\circ\text{C}} = C_{25^\circ\text{C}} + C_{25^\circ\text{C}} * 1.94 / 100 * (30^\circ\text{C} - 25^\circ\text{C})$
 $= 1.413 \text{ mS} + 1.413 * 1.94 / 100 * 5 = 1.55 \text{ approx.}$

Caution: Conductivity cell of 0.1 K should both be standardized with 0.1 M KCl solution as the conductivity of this solution falls beyond its range.

(ii) Turbidity:

A sample is turbid if it contains suspended matter that interferes with passage of light through the water or in which visual depth is restricted. The turbidity can be caused by colloidal particles to coarse particles, depending on degree of turbulence. This parameter is important due to aesthetics, filterability of sample and effectiveness of the disinfection process for killing pathogens from water.

Standard unit of turbidity

Currently turbidity measurements are done by the standard nephelometry procedure and turbidity is represented in Nephelometric Turbidity Unit (NTU). This procedure is conducted using formazin standard.

Method of determination (2130 B Nephelometric Method)

In the nephelometric instrument, a light source illuminates the source and one or more photoelectric electric detectors are used with a readout device to indicate the intensity of scattered light at right angles to the path of the incident light. This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the intensity of scattered light, the higher is the turbidity of the sample. Formazin polymer is used as the primary standard reference suspension. The turbidity of a specified concentration of formazin suspension is defined as 4000 NTU. During use of formazin standard, 1 NTU = 1 Jackson Candle Turbidity Unit (Jackson Candle was used earlier to measure turbidity of different samples). Turbidities as low as 0.02 NTU can be determined through this procedure and samples with turbidity >40 NTU are diluted with turbidity-free water until values lie in the range of 30 to 40 NTU. The turbidity is determined by multiplying with dilution factor

Experimental Procedure

(i) pH

1. Calibrate pH meter and record pH of the sample.

(ii) Conductivity 1. For conductivity measurement, first determine resistance. For this, first rinse conductivity cell with at least three portions of 0.01M KCl solution and measure resistance (R). Adjust temperature to 25°C.

(ii) Conductivity

1. For conductivity measurement, first determine resistance. For this, first rinse conductivity cell with at least three portions of 0.01M KCl solution and measure resistance (R). Adjust temperature to 25°C

2. Using resistance and temperature information, calculate cell constant, i.e., C (1/cm) (note that sometimes, conductivity meters indicate conductivity values directly):

$$C(1/cm) = (0.001412 * RKCl) * [1 + 0.0191 * (t - 25)]$$

where, RKCl = measured resistance, ohms and t = observed temperature (°C)

3. For internal calibration of cell, rinse cell with 0.01M KCl and adjust meter to read 1412 µmho/cm. This procedure automatically adjusts cell constant internal to the meter.

4. For measuring conductivity of an unknown sample, repeat step 3 and note down resistance and temperature upto ±0.1°C.

5. When sample resistance is measured, conductivity at 25°C is given by this equation:

$$k(\mu\text{mho/cm}) = (1/R_m) * (1000000 * C) / [1 + 0.0191 * (t - 25)]$$

where, C = cell constant (1/cm)

t = observed temperature (°C)

R_m = measured resistance, ohms and

6. When sample conductivity is measured without internal temperature compensation, conductivity at 25°C is given by this equation:

$$k (\mu\text{mho/cm}) = (k_m) / [1 + 0.0191 * (t - 25)]$$

where, k_m is measured conductivity at t °C and other units are defined as above

(iii) Turbidity

Reagents:

a. Stock primary standard formazin suspension:

1. Solution I—Dissolve 1 g hydrazine sulfate [(NH₂)₂.H₂SO₄] in distilled water and dilute to 100 mL in a volumetric flask. CAUTION: Hydrazine sulfate is a carcinogen; avoid inhalation, ingestion, and skin contact. Formazin suspensions can contain residual hydrazine sulfate.
2. Solution II—Dissolve 10.00 g hexamethylenetetramine, (CH₂)₆N₄, in distilled water and dilute to 100 mL in a volumetric flask.
3. In a flask, mix 5.0 mL Solution I and 5.0 mL Solution II. Let it stand for 24 h at 25 ± 3°C, which results in a 4000-NTU suspension. Transfer the stock suspension to an amber glass or other UV-light-blocking bottle for storage. The stock suspension is stable for up to 1 year when properly stored. Make dilutions from this stock suspension.

b. Diluted turbidity suspensions:

Dilute 4000 NTU primary standard suspension with high quality dilution water. Prepare immediately and discard after the use.

Measurement:

1. Calibrate nephelometer as per the instructions.
2. For unknown sample, first agitate the sample and wait until air bubbles disappear and then pour the well-mixed sample into cell. Make sure to release air bubble otherwise it might interfere with readings. Read turbidity from the instrument display.
3. Interpretation of results: Report turbidity readings as follows:

| Reporting of turbidity values | |
|-------------------------------|---------------------------|
| Turbidity range (NTU) | Report to the nearest NTU |
| 0-1.0 | 0.05 |
| 1-10 | 0.1 |
| 10-40 | 1 |
| 40-100 | 5 |
| 100-400 | 10 |
| 400-1000 | 50 |
| >1000 | 100 |

Answer these questions:

1. Why pH is an important parameter in environmental engineering? Did you find any different in pH of two samples? Why or why not?
2. How does conductivity represent ions in solution?
3. Discuss the nature of materials causing turbidity in (a) river water during monsoon period, (b) polluted river water and (c) domestic wastewater.

DETERMINATION OF TOTAL ,SUSPENDED, AND DISSOLVED SOLIDS

AIM

The aim of the experiments is determination of total, suspended and dissolved solids in water.

APPARATUS REQUIRED

1. Balance 2. Beaker 3. Measuring Cylinder 4. Filter paper/ or Gooch Crucible 5. Funnel 6. Dropper

PROCEDURE:

(a) Measurement of Total Solids (TS)

(1) Take a clear dry glass beaker of 150 ML capacity (which was kept at 103°C in an oven for 1 hour) and put appropriate identification mark on it. Weight the beaker and note the weight.

(2) Pour 100ml. of the thoroughly mixed sample, measured by the measuring cylinder, in the beaker.

(3) Place the beaker in an oven maintained at 103°C for 24 hours. After 24 hours, when whole of the water has evaporated, cool the beaker and weight. Find out the weight of solids in the beaker by subtracting the weight of the clean beaker determined in step (1)

(4) Calculate total solids (TS) as follows:

Total Solids in water = Difference of weight of the beakers / Volume of sample X 1000

(b) Measurement of Total Dissolved Solids (TDS)

(1) Same as above (step 1 of total solids).

(2) Take a 100 ml. of sample and filter it through a double layered filter paper or a Gooch Crucible and collect the filtrate in a beaker.

(3) Then repeat the same procedure as in steps (3) and (4) of the total solids determination and determine the dissolved solids contents as follows:

CALCULATION:

Dissolved solids, TDS (mg/l) = mg of solids in the beaker / (volume of sample) x 1000

Also total solid (TS) = Suspended Solids + Total dissolved Solids (TDS)

NOTE:- See the experiment on the link below:-

https://www.youtube.com/watch?v=GJSe_Deo-_0&ab_channel=NCTEL

Objective: Measure (1) Total hardness and (2) Calcium hardness using dye indicators

Background:

Hard Water:

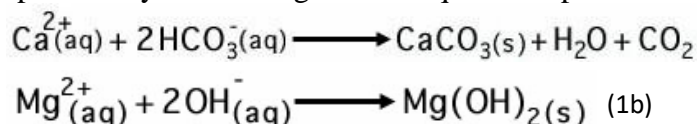
Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam and that also produce scale in water pipes, heaters, boilers and other units in which the temperature of water is increased. Hard water are appropriate for human consumption similar to that as soft waters, however it produces adverse actions with soap and thus their use for cleaning purposes is unsatisfactory and thus their removal from water is required. Hardness of waters varies from place to place. In general, surface waters are softer than ground waters. Waters are commonly classified based on degree of hardness (Table 1):

Table 1. Classification of hardness types

| Hardness (mg/L) | Degree of hardness |
|-----------------|--------------------|
| 0-75 | Soft |
| 75-100 | Moderately hard |
| 150-300 | Hard |
| >300 | Very hard |

Hardness:

Hardness is caused by polyvalent metallic cations, though the divalent cations, such as calcium and magnesium cations are usually the predominant cause of hardness. In addition, hardness is also caused by Fe²⁺ and Mn²⁺ ions. For example, when hard water is heated, Ca²⁺ ions react with bicarbonate (HCO₃⁻) ions to form insoluble calcium carbonate (CaCO₃) (Eq. 1). This precipitate, known as *scale*, coats the vessels in which the water is heated, producing the mineral deposits on your cooking dishes. Equation 2 presents magnesium hardness.



Total hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L. When hardness (numerically) is greater than the sum of carbonate and bicarbonate alkalinity, amount of hardness equivalent to the total alkalinity is called “**Carbonate hardness**”.

$$\text{Carbonate hardness (mg/L)} = \text{Alkalinity} \quad (2\text{a})$$

When alkalinity > Total hardness:

$$\text{Carbonate hardness (mg/L)} = \text{Total hardness} \quad (2\text{b})$$

The amount of hardness in excess of this is called “**Non-carbonate hardness (NCH)**”. These are associated with sulfate chloride, and nitrate ions. It is calculated using Eq (2c):

$$\text{NCH (mg/L)} = \text{Total hardness} - \text{Carbonate hardness} \quad (2\text{c})$$

Determination of Hardness:

Hardness is expressed as mg/L CaCO₃. The first method is calculation based method and the second method is titration method using EDTA.

(i) Calculation method

For this method, concentration of cations should be known and then all concentrations are expressed in terms of CaCO₃ using **Eq. 3:**

$$\text{Hardness (in mg/L as CaCO}_3) = [\text{M}^{2+} (\text{in mg/L}) \times 50] / (\text{E.Wt. of M}^{2+}) \quad (3)$$

Where: M²⁺ = mass of divalent ions (mg/L) and E.Wt. = Equivalent weight of divalent ions (g/mole)

Example: If in a sample, 15 mg/L Ca^{2+} are present, hardness is given by

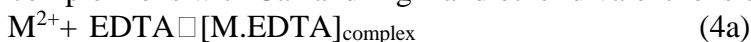
$$\text{Hardness (in mg/L as CaCO}_3) = [\text{mass of Ca}^{2+} \text{ (in mg/L)} \times 50] / (\text{E.Wt. of Ca}^{2+})$$

Here, E.Wt. of $\text{Ca}^{2+} = (40\text{g/mole})/2 = 20 \text{ g/mole}$

So, Hardness due to calcium ions = $[15 \text{ mg/L} \times 50] / (20) = 37.5 \text{ mg/L CaCO}_3$

(ii) EDTA Titrimetric Method

This method uses ethylenediaminetetracetic acid (EDTA), chelating agents, which forms complex ions with Ca^{2+} and Mg^{2+} and other divalent ions causing hardness (Eq. 4a):



The successful use of EDTA for determining hardness depends on presence of an indicator which can show presence of excess EDTA in solution or when all the ions present in solution have been complexed. Eriochrome Black T (EBT) (blue color solution) serves as an excellent indicator to show when all hardness ions have been consumed. When small amount of EBT is added to hard water with $\text{pH} > 10$, it combines with Ca^{2+} and Mg^{2+} ions to form weak complex ions (wine-red color solution) (Eq. 4b):



During the titration with EDTA, all free hardness ions are complexed as per Eq. 4a and subsequently, EDTA disrupts the wine red complex as it can form a stable complex with the hardness ions. At this stage, solution color changes from red wine color to blue color, indicating the end of the titration.

Lab Procedure:

Reagents: Buffer solution; EDTA Titrant; EBT

1. Measure Ca-Hardness and Total Hardness by titration as described below. **Use a different sample for each measurement.**
2. **Total Hardness:** Take 100 ml of the sample and add 2 ml buffer solution in it and add 2- 3 drops of Black T. Titrate it with standard EDTA solution (with continuous stirring) until the last reddish colour disappears. At the end point the solution turns blue. **Note down the volume used.** Calculate Hardness as follows:
Hardness (in mg/L as CaCO_3) = $(V \times N \times 50 \times 1000) / (SV) \quad (5)$
Where: V = volume of titrant (mL); N = normality of EDTA; 50 = equivalent weight of CaCO_3 ; SV = sample volume (mL)
3. **Ca-Hardness:** Take 50 ml of the sample and add 1 ml Sodium Hydroxide solution (8%) in it and add pinch of Mercurex Powder. Titrate with standard EDTA solution until the light pink colour of solution converts into light blue color.

Answer these questions also:

1. Among finished drinking water, raw wastewater and de-ionized water, which water is expected to have the highest carbonate hardness and why?
2. A sample has 50mg/L Ca^{2+} , 150mg/L Mg^{2+} , 50 mg/L Na^+ , 20 mg/L Cl^- and 100 mg/L glucose. Calculate its total hardness, carbonate and non-carbonate hardness?

Reference Materials:

AWWA, WEF, APHA, 1998, Standard Methods for the Examination of Water and Wastewater (Methods: 2340 C. EDTA Titrimetric Method)

Sawyer, C.N., McCarty, P.L., and Parkin, G.F. 2000. *Chemistry for Environmental Engineering*

4th Edition. Tata McGraw-Hill Publishing Company Limited.

Jar Test for Determining Optimum Coagulant Dosage

Aim

To determine the optimum coagulant dosage for clarifying the given sample of water by using alum as the coagulant and performing the jar test experiment.

Principle

Coagulants are used in water treatment plants to remove natural suspended and colloidal matter, to remove material which do not settle in plain sedimentation, and to assist in filtration. Alum $[Al_2S(SO_4)_3 \cdot 18H_2O]$ is the most widely used coagulant. When alum solution is added to water, the molecules dissociate to yield SO_4^{2-} and Al^{3+} . The +ve species combine with negatively charged colloidal to neutralise part of the charge on the colloidal particle. Thus, agglomeration takes place. Coagulation is a quite complex phenomenon and the coagulant should be distributed uniformly throughout the solution. A flash mix accomplishes this.

Jar test is simple device used to determine this optimum coagulant dose required. The jar test, device consists of a number of stirrers (4 to 6) provided with paddles. The paddles can be rotated with varying speed with the help of a motor and regulator. Samples will be taken in jars or beakers and varying dose of coagulant will be added simultaneously to all the jars. The paddles will be rotated at 100 rpm for 1 minute and at 40 rpm for 20 to 30 minutes, corresponding to the flash mixing and slow mixing in the flocculator of the treatment plant. After 30 minutes settling, supernatant will be taken carefully from all the jars to measure turbidity. The dose, which gives the least turbidity, is taken as the optimum coagulant dose.

Apparatus

Jar test apparatus

1. Glass beakers
2. Pipette
3. Nephelometer
4. pH meter
5. Reagents (click to check the preparation of reagents)
6. Alum solution (1mL containing 10 mg of alum)
7. Lime
8. Acid/alkali

Procedure

1. Take 1-litre beakers and fill them with sample up to the mark.
2. Keep each beaker below each paddle and lower the paddles, such that each one is about 1cm above the bottom.
3. Find the pH of the sample and adjust it to 6 to 8.5.
4. Pipette 1, 2, 3, 4, 5, 6 mL of the alum solution into the test samples.
5. Immediately run the paddles at 100 rpm for 1 minute.

6. Reduce the speed to 30-40 rpm and run at this rate for 30 minutes.
7. Stop the machine, lift out the paddles and allow to settle for 30 minutes.
8. Find the residual turbidity of the supernatant using nephelometer.
9. Plot a graph with alum dosage along x-axis and turbidity along y-axis.
10. The dosage of alum, which represents least turbidity, gives Optimum Coagulant Dosage (O.C.D.).
11. Repeat steps 1-10 with higher dose of alum, if necessary.

Observation

| <i>Trial no.</i> | <i>Alum dosage in mg/L</i> | <i>Turbidity in NTU</i> |
|------------------|----------------------------|-------------------------|
| | | |
| | | |
| | | |
| | | |
| | | |

Result Optimum coagulant dosage =

Chemical Oxygen Demand

OBJECTIVE: To measure Chemical Oxygen Demand.

BACKGROUND AND PRINCIPLE:

The chemical oxygen demand (COD) determines the amount of oxygen required for chemical oxidation of organic matter using a strong chemical oxidant, such as, potassium dichromate under reflux conditions. This test is widely used to determine:

- a) Degree of pollution in water bodies and their self-purification capacity,
- b) Efficiency of treatment plants,
- c) Pollution loads, and
- d) Provides rough idea of Biochemical oxygen demand (BOD) which can be used to determine sample volume for BOD estimation.

The limitation of the test lies in its inability to differentiate between the biologically oxidizable and biologically inert material and to find out the system rate constant of aerobic biological stabilization.

Most of the organic matters are destroyed when boiled with a mixture of potassium dichromate and sulphuric acid producing carbon dioxide and water. A sample is refluxed with a known amount of potassium dichromate in sulphuric acid medium and the excess of dichromate is titrated against ferrous ammonium sulphate. The amount of dichromate consumed is proportional to the oxygen required to oxidize the oxidizable organic matter.

SELECTION OF METHODS

There are two methods available for COD determination namely open reflux and closed reflux.

Open Reflux Principle:

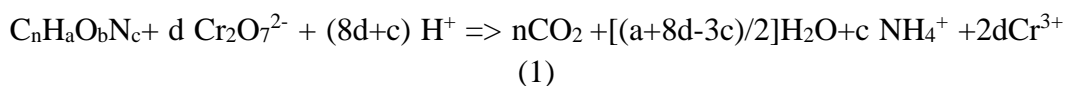
- Suitable for a wide range of wastes with a large sample size.
- Due to its higher oxidizing ability dichromate reflux method is preferred over other procedures using other oxidants (e.g. potassium permanganate).
- Oxidation of most organic compounds is up to 95-100% of the theoretical value.

Closed Reflux Principle:

- This method is conducted with ampoules and culture tubes with pre-measured reagents.
- Measurement of sample volume and reagent volume are critical.

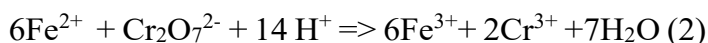
- This method is economical in the use of metallic salt reagents and generate smaller quantity of hazardous wastes.
- Volatile organic compounds (VOC) gets completely oxidized in a closed system than the open because of longer contact time with oxidants.

Chemical Reactions:



Here $d = (2n/3) + (a/6) - (b/3) - (c/2)$

During experiment, excess dichromate concentration is determined by titrating it with ferrous ammonium sulfate (FAS). The reaction is given by:



Here $d = (2n/3) + (a/6) - (b/3) - (c/2)$

SAMPLING AND HANDLING REQUIREMENTS

| S . No | Determination | Container | Preservation Technique | Min Vol ,ml | Maximum storage Recommended | Regulatory |
|--------|---------------|----------------|--|-------------|-----------------------------|------------|
| 1. | COD | Plastic ,Glass | Analyze as soon as possible , or add H ₂ SO ₄ to pH < 2; refrigerate | 100 | 7 days | 28 d |

REAGENTS:

- Standard Potassium dichromate (K₂Cr₂O₇) digestion solution, 0.01667M:

Add to about 500 mL distilled water 4.903 g K₂Cr₂O₇, primary standard grade, previously dried at 150°C for 2 h, 167 mL conc. H₂SO₄, and 33.3 g HgSO₄. Dissolve, cool to room temperature, and dilute to 1000 mL.

- Sulfuric acid reagent:

Add H₂SO₄ at the rate of 5.5 g Ag₂SO₄/kg H₂SO₄ or 10.12 g silver sulphate/L H₂SO₄. Let stand 1 to 2 d to dissolve and mix. This accelerates the oxidation of straight-chain aliphatic and aromatic compounds.

(1 Kg = 543.47826 mL of H₂ SO₄ and take 20.24 g of Ag₂SO₄ to 2 L of H₂ SO₄ or 22.264 g of Ag₂SO₄ to 2.2 L of H₂ SO₄)

➤ Ferriin Indicator solution:

This indicator is used to indicate change in oxidation-reduction potential of the solution and indicates the condition when all dichromate has been reduced by ferrous ion. It gives a very sharp brown color change which can be seen in spite of blue color generated by the Cr^{3+} ions formed on reduction of the dichromate.

➤ Standard ferrous ammonium sulfate titrant (FAS), approximately 0.10M:

Dissolve 39.2 g $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ in distilled water. Add 20 mL conc. H_2SO_4 , cool, and dilute to 1000 mL. Standardize solution daily against standard $\text{K}_2\text{Cr}_2\text{O}_7$ digestion solution as follows: Pipet 5.00 mL digestion solution into a small beaker. Add 10 mL reagent water to substitute for sample. Cool to room temperature. Add 1 to 2 drops diluted Ferriin indicator and titrate with FAS titrant.

$$\text{Molarity of FAS solution} = [\text{V}_{\text{K}_2\text{Cr}_2\text{O}_7} \times 0.1] / (\text{V}_{\text{FAS}})$$

Where: $\text{V}_{\text{K}_2\text{Cr}_2\text{O}_7}$ = volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (mL); V_{FAS} = volume of FAS (mL)

PROCEDURE:

1. Wash culture tubes and caps with 20% H_2SO_4 before using to prevent contamination.
2. Place sample (2.5 mL) in culture tube and Add $\text{K}_2\text{Cr}_2\text{O}_7$ digestion solution (1.5 mL).
3. Carefully run sulphuric acid reagent (3.5 mL) down inside of vessel so an acid layer is formed under the sample-digestion solution layer and tightly cap tubes or seal ampules, and invert each several times to mix completely.
4. Place tubes in block digester preheated to 150°C and reflux for 2 h behind a protective shield.
5. Cool to room temperature and place vessels in test tube rack. Some mercuric sulfate may precipitate out but this will not affect the analysis.
6. Add 1 to 2 drops of Ferriin indicator and stir rapidly on magnetic stirrer while titrating with standardized 0.10 M FAS.
7. The end point is a sharp color change from blue-green to reddish brown, although the blue green may reappear within minutes.
8. In the same manner reflux and titrate a blank containing the reagents and a volume of distilled water equal to that of the sample.
9. COD is given by

$$\text{COD (mg O}_2\text{ /L)} = [(A-B) \times M \times 8000] / (\text{V}_{\text{sample}})$$

Where: A = volume of FAS used for blank (mL)
B = volume of FAS used for sample (mL)
M = molarity of FAS
8000 = milli equivalent weight of oxygen (8) \times 1000 mL/L.

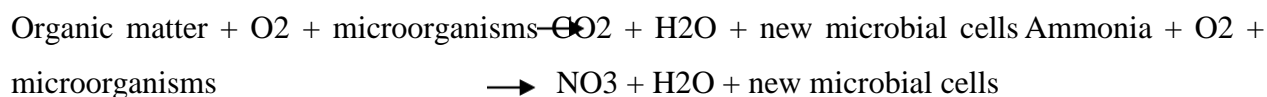
ANSWER THESE QUESTIONS ALSO:

1. Why do the COD analysis and BOD analysis give different results for the same waste?
2. What could be inferred from the following samples concerning the relative ease of

biodegradability: Sample A (5-d BOD/COD=24/30) and Sample B (5-d BOD/COD=10/50)?

Aim: Determine biological oxygen demand (BOD) of given sewage samples.

Introduction: The amount of oxygen, taken up by the microorganisms that decompose the organic waste matter in wastewater is known as biological oxygen demand or biochemical oxygen demand. Therefore it is used to measure the amount of certain type of organic water pollution BOD is calculated by keeping a sample of water containing a known amount of oxygen for five days at 20°C. The oxygen content is measured again and BOD is calculated. A high BOD indicates the presence of a large number of microorganisms which indicates a high level of pollution in wastewater. Oxygen demand is associated with the biodegradation of the carbonaceous portion of wastes and oxidation of nitrogen compounds such as ammonia. The following equations simplify the process of biodegradation:



Requirements: Incubation bottle 300mL volume; Air compressor, 20°C incubator, OD measure reagents.

Procedure:

1. Prepare BOD dilutions. 5 mL sample in 300 mL BOD bottle, fill up with dilution water; 15 mL sample in 300 mL BOD bottle, fill up with dilution water; 20 mL sample in 300 mL BOD bottle, fill up with dilution water.
2. Take 300 mL sample in BOD bottle. Prepare two sets of this sample. Keep one set for DO analysis for day 0 (i.e., Sample0Day) and another sample in BOD incubator for 5 days at 20°C (Sample5Day).
3. Measure DO in different samples at t=0.
4. Incubate samples in 20°C for 5 days.
5. Come back in the lab after 5 days and record dissolved oxygen.
6. Record data in following manner.

| Bottle no. | Wastewater sample (mL) | Initial DO (mg/L) (DO ₀) | DO at 5-day (mL) (DO ₅) |
|------------|------------------------|--------------------------------------|-------------------------------------|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |

Calculations:

Calculate 5-day BOD value of the sample at 20°C:

$$t\text{-day BOD} = [\text{DO}_t - \text{DO}_0] / (P) \quad (1)$$

Where P = Dilution factor = 300mL / (sample volume in mL)

Observation table:

| BOD Level in mg/liter | Water Quality |
|-----------------------|---|
| 1 - 2 | Very Good: There will not be much organic matter present in the water supply. |
| 3 - 5 | Fair: Moderately Clean |
| 6 - 9 | Poor: Somewhat Polluted - Usually indicates that organic matter present and microorganisms are decomposing that waste. |
| 100 or more | Very Poor: Very Polluted - Contains organic matter. |

Break Point Chlorination

Chloramines / Combined Chlorine

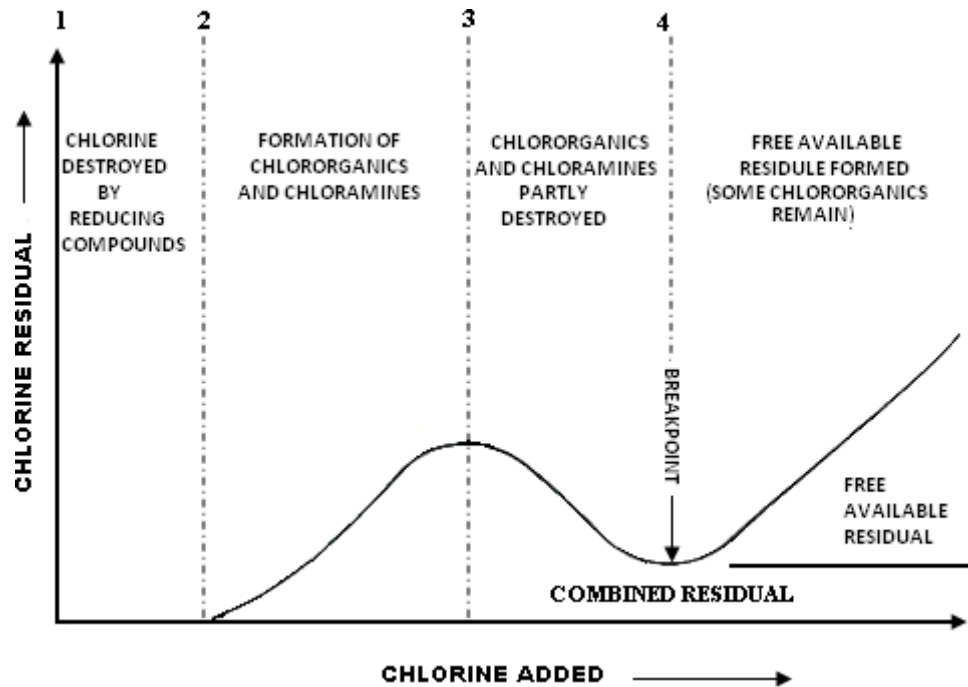
If you smell “chlorine”, coming from your pool, what you really smell are combined forms of chlorine, also called chloramines. Chloramines are chemical compounds formed by chlorine combining with nitrogen containing contaminants in the pool water. These are still disinfectants, but they are 40 to 60 times less effective than free available chlorine. Contaminants come from swimmer wastes such as sweat, urine, body oil, etc. Therefore, requiring all bathers to take a warm, soapy water shower is a good idea.

Three types of chloramines can be formed in water - monochloramine, dichloramine, and trichloramine. Monochloramine is formed from the reaction of hypochlorous acid with ammonia. Monochloramine may then react with more hypochlorous acid to form dichloramine. Finally, the dichloramine may react with hypochlorous acid to form a trichloramine. Trichloramines cause the “chlorine” smell and hang in the air directly above the pool water level, often causing competitive or frequent swimmers to have asthma like symptoms. High levels of chloramines will also cause corrosion to surfaces and equipment in the pool area. The trichloramines are especially irritating to the eyes, nose and lungs.

Chloramines can usually be eliminated from the pool water by performing breakpoint chlorination with chlorine or super oxidation with a non-chlorine oxidizer. Ultraviolet systems and ozone systems are effective at reducing chloramines in pools.

Breakpoint chlorination

Break point chlorination is adding enough chlorine to eliminate problems associated with combined chlorine. Specifically, breakpoint chlorination is the point at which enough free chlorine is added to break the molecular bonds, specifically the combined chlorine molecules, ammonia or nitrogen compounds. It takes a ratio of chlorine to ammonia atoms of 7.6 to 1 to reach breakpoint, other contaminants (i.e., bacteria, algae) are also present that must be oxidized, so 10 times the amount of combined chlorine must be added. When sufficient free chlorine (FC) is added to pool water, the inorganic chloramines are converted to dichloramine, then to nitrogen trichloride, and then to nitrogen gas. Any excess chlorine leftover will become the chlorine residual (FC). The graph below shows what happens when chlorine (either chlorine gas or a hypochlorite) is added to water. First (between points 1 and 2), the water reacts with reducing compounds in the water, such as hydrogen sulfide. These compounds use up the chlorine, producing no chlorine residual.



Between points 2 and 3, the chlorine reacts with organics and ammonia naturally found in the water. Some combined chlorine residual is formed – chloramines.

Between points 3 and 4, the chlorine will break down most of the chloramines in the water, actually lowering the chlorine residual.

Finally, the water reaches the breakpoint, shown at point 4. The breakpoint is the point at which the chlorine demand has been totally satisfied - the chlorine has reacted with all reducing agents, organics, and ammonia in the water. When more chlorine is added past the breakpoint, the chlorine reacts with water and forms hypochlorous acid in direct proportion to the amount of chlorine added.

The combined chlorine (CC) level is calculated by subtracting the free chlorine (FC) from the total chlorine (TC) in the pool/spa water. Rule 410 IAC 6-2.1-30(o) 2 requires testing of the pool/spa water for combined levels at least twice a week.

Rule 410 IAC 6-2.1-30(e) requires “The pool water shall be superchlorinated to breakpoint or superoxidized with a nonchlorine oxidizer, when the pool test kit reveals a

combined chlorine (chloramine) concentration of five-tenths (0.5) parts per million (ppm) or greater.” However, studies have shown that swimmers find pool water the most enjoyable if more than 85% of the total chlorine is free chlorine. Therefore, the Environmental Public Health Staff recommends superchlorination when the combined chlorine concentration is 0.2 ppm or greater (a total chlorine of 1.2 ppm and a free chlorine of 1.0 ppm provides 83% of total chlorine as free chlorine).

Note: Pools using bromine as a sanitizer must also perform breakpoint superchlorination using chlorine. Like chlorine, bromine combines with organic impurities to form combined bromine and bromamines.

Calculating Amount of Chemical to Achieve Breakpoint Chlorination

The DPD test does not measure combined chlorine (CC) directly, it measures free chlorine (FC) in Step 1 and total chlorine (TC) in Step 2. Total Chlorine is the sum of free chlorine and combined chlorine. Therefore, combined chlorine is the difference between total chlorine and free chlorine. $CC = TC - FC$.

The first step in determining the necessity of a shock treatment is to determine the level of combined chlorine.

Using the D.P.D. testing kit, test for free chlorine (FC) and total chlorine (TC). After completing the water test, you subtract the free chlorine reading from the total available chlorine reading, the result indicates the combined chlorine (CC) or chloramine level in the pool water.

For example:

Combined Chlorine = Total Chlorine - Free Chlorine

2.3 ppm (TC) measured from test kit - 1.5 ppm (FC) measured from test kit = 0.8 ppm CC.

If the water has no chloramines, the answer to the subtraction will be zero (0) and a shock treatment is not needed. This is a desirable level. After determining the level of combined chlorine in the pool water, the pool operator must determine the breakpoint chlorination for that value.

The breakpoint chlorination value is 10 times the combined chlorine (CC) level.

For example: 0.8 ppm (CC) from the above example $\times 10 = 8$ ppm of chlorine to achieve breakpoint.

Taking into account the free chlorine already in the pool, chlorine will have to be added to the level of 8 ppm.

Principle

Water to be tested is diluted serially and inoculated in lactose broth, coliforms if present in water utilizes the lactose present in the medium to produce acid and gas. The presence of acid is indicated by the 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

Presumptive test

Confirmatory test

Completed test

Presumptive test

The presumptive test is a screening test to sample water for the presence of coliform organisms.

If the presumptive test is negative, no further testing is performed, and the water source is considered microbiologically safe.

If the presumptive test is negative, no further testing is performed, and the water source is considered microbiologically safe. If, however, any tube in the series shows acid and gas, the water is considered unsafe and the confirmed test is performed on the tube displaying a positive reaction.

The method of the presumptive test varies for treated and untreated water.

Requirements

Medium: Lactose broth or MacConkey broth or Lauryl tryptose (lactose) broth

Glasswares: Test tubes of various capacities (20ml, 10ml, 5ml), Durham tube

Others: Sterile pipettes

Preparation of the Medium

Prepare medium (either MacConkey broth or lactose broth) in single and double strength concentrations.

For untreated or polluted water :

Dispense the double strength medium in 10 tubes (10mL in each tube) and single strength medium in 5 tubes (10 mL in each tube)and add a Durham tube in an inverted position.

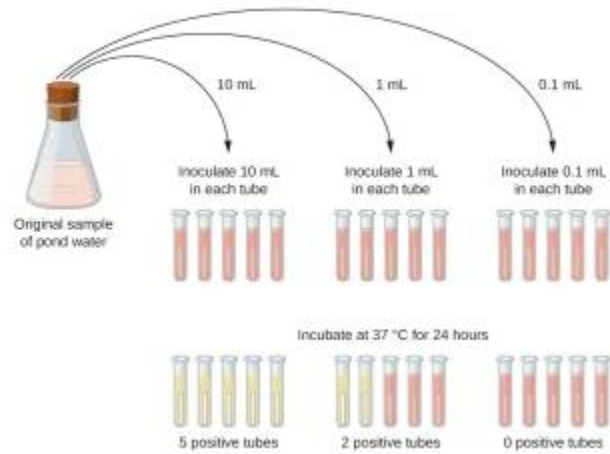
For treated water:

Dispense the double strength medium in 5 tubes (10mL in each tube) and 50 mL single strength medium in 1 bottle and add a Durham tube in an inverted position.

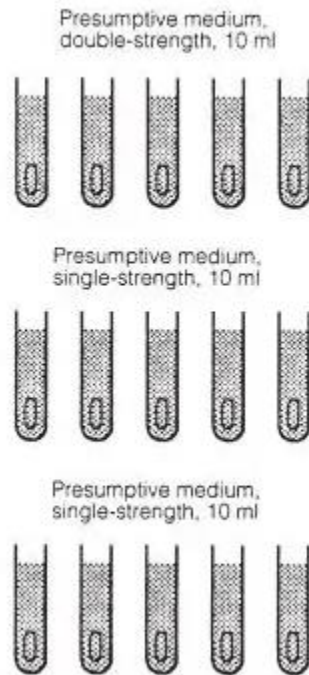
Examine the tubes to make sure that the inner vial is full of liquid with no air bubbles.

Sterilize by autoclaving at 15 lbs pressure (121°C) for 15 minutes.

Procedure of MPN test



For untreated (polluted) water



MPN Water Testing

Take 5 tubes of double strength and 10 tubes of single strength for each water sample to be tested.

Using a sterile pipette add 10 mL of water to 5 tubes containing 10 mL double strength medium.

Similarly, add 1 mL of water to 5 tubes containing 10 mL single strength medium and 0.1 mL water to the remaining 5 tubes containing 10 mL single strength medium.

Incubate all the tubes at 37°C for 24 hrs. If no tubes appear positive re-incubate up to 48 hrs.

Compare the number of tubes giving a positive reaction to a standard chart and record the number of bacteria present in it.

For example, a water sample tested shows a result of 3–2–1 (3 × 10 mL positive, 2 × 1 mL positive, 1 × 0.1 mL positive) gives an MPN value of 17, i.e. the water sample contains an estimated 17 coliforms per 100 ml

To view the full table download the PDF file from the link given in the reference.

For treated (unpolluted) water

Take 1 tube of single strength (50mL) and 5 tubes of double strength (10mL) for each water sample to be tested.

Using a sterile pipette add 50 mL of water to the tubes containing 50 mL single strength medium.

Similarly, add 10 mL of water to 5 tubes containing 10 ml double strength medium.

Incubate the tubes at 37°C for 24 hrs. If no tubes appear positive re-incubate up to 48 hrs.

Compare the number of tubes giving a positive reaction to a standard chart and record the number of bacteria present in it.

For example, a water sample tested shows a result of 1-4 (1 × 50 mL positive, 4 × 10 mL positive) gives an MPN value of 16, i.e. the water sample contains an estimated 16 coliforms per 100 mL.

MPN values per 100 ml of sample and 95% confidence limits for various combinations of positive and negative results (when five 10-ml, five 1-ml and five 0.1 ml test portions are used)

| No. of tubes giving a positive reaction : | | | MPN (per 100 ml) | 95% confidence limits | |
|---|-----------|-------------|------------------|-----------------------|-------|
| 5 of 10ml | 5 of 1 ml | 5 of 0.1 ml | | Lower | Upper |
| 0 | 0 | 0 | <2 | <1 | 7 |
| 0 | 1 | 0 | 2 | <1 | 7 |
| 0 | 2 | 0 | 4 | <1 | 11 |
| 1 | 0 | 0 | 2 | <1 | 7 |
| 1 | 0 | 1 | 4 | <1 | 11 |
| 1 | 1 | 0 | 4 | <1 | 11 |
| 1 | 1 | 1 | 6 | <1 | 15 |
| 2 | 0 | 0 | 5 | <1 | 13 |
| 2 | 0 | 1 | 7 | 1 | 17 |
| 2 | 1 | 0 | 7 | 1 | 17 |
| 2 | 1 | 1 | 9 | 2 | 21 |
| 2 | 2 | 0 | 9 | 2 | 21 |
| 2 | 3 | 0 | 12 | 3 | 28 |
| 3 | 0 | 0 | 8 | 1 | 19 |
| 3 | 0 | 1 | 11 | 2 | 25 |
| 3 | 1 | 0 | 11 | 2 | 25 |
| 3 | 1 | 1 | 14 | 4 | 34 |
| 3 | 2 | 0 | 14 | 4 | 34 |
| 3 | 2 | 1 | 17 | 5 | 46 |

Confirmatory Test

- Some microorganisms other than coliforms also produce acid and gas from lactose fermentation. In order to confirm the presence of coliform, a confirmatory test is done.
- From each of the fermentation tubes with positive results transfer one loopful of medium to:
 - mL lactose-broth or brilliant green lactose fermentation tube,
 - to an agar slant and
 - mL tryptone water.
- Incubate the inoculated lactose-broth fermentation tubes at 37°C and inspect gas formation after 24 ± 2 hours. If no gas production is seen, further incubate up to a maximum of 48 ± 3 hours to check gas production.
- The agar slants should be incubated at 37°C for 24± 2 hours and Gram-stained preparations made from the slants should be examined microscopically.
- The formation of gas in lactose broth and the demonstration of Gram-negative, non-spore-forming bacilli in the corresponding agar indicates the presence of a member of the coliform group in the sample examined.

- The absence of gas formation in lactose broth or the failure to demonstrate Gram-negative, non-spore-forming bacilli in the corresponding agar slant constitutes a negative test (absence of coliforms in the tested sample).

Tryptone Water Test

Incubate the tryptone water at $(44.5 \pm 0.2^\circ\text{C})$ for 18-24 hours

Following incubation, add approximately 0.1mL of Kovacs reagent and mix gently.

The presence of indole is indicated by a red color in the Kovacs reagent, forming a film over the aqueous phase of the medium.

- a. Confirmatory tests positive for indole, growth, and gas production show the presence of thermotolerant *E. coli*.
- b. Growth and gas production in the absence of indole confirm thermotolerant coliforms.

Completed Test

Since some of the positive results from the confirmatory test may be false, it is desirable to do completed tests. For this inoculum from each positive tube of the confirmatory test is streaked on a plate of EMB or Endo agar.

In this process, a loopful of a sample from each positive BGLB tube is streaked onto selective medium like Eosin Methylene Blue agar or Endo's medium. One plate each is incubated at 37°C and another at $44.5 \pm 0.2^\circ\text{C}$ for 24 hours.

High temperature incubation (44.5 ± 0.2) is for detection of thermotolerant *E. coli*.

Following incubation, all plates are examined for the presence of typical colonies.

Coliforms produce colonies with a greenish metallic sheen which differentiates it from non-coliform colonies (show no sheen). The presence of typical colonies on high temperature (44.5 ± 0.2) indicates the presence of thermotolerant *E. coli*.

Advantages of MPN

1. Ease of interpretation, either by observation or gas emission
2. Sample toxins are diluted
3. Effective method of analyzing highly turbid samples such as sediments, sludge, mud, etc. that cannot be analyzed by membrane filtration.

Disadvantages of MPN

1. It takes a long time to get the results

2. Results are not very accurate
3. Requires more hardware (glassware) and media
4. Probability of false positives

QUIZ

1. The term 'Sullage' refers to:
 - a) Fresh wastewater
 - b) Septic wastewater
 - c) Wastewater from kitchen, laundry
 - d) Toxic wastewater
2. Wastewater can become septic by the loss of:
 - a) Dissolved oxygen content
 - b) Carbon content
 - c) Organic compounds
 - d) Water content
3. Which one of the below is not an attribute of drinking water?
 - a) Aesthetics
 - b) Economic
 - c) Safety
 - d) Source
4. The extent of water treatment depends on how many factors?
 - a) 5
 - b) 2
 - c) 3
 - d) 4
5. One of the major objectives of water treatment plants is the removal of turbidity.
 - a) True
 - b) False
6. What is added to the water treatment tank to settle the colloidal particles?
 - a) Alum
 - b) Alum and lime
 - c) Lime
 - d) Potash
7. Disinfection of water in our country is mainly done by _____
 - a) Oxygenation
 - b) Hydration
 - c) Chlorination
 - d) Filtration
8. Which minerals and in what form are present in ground water?
 - a) Fe & Mn in Ferrous and Manganous
 - b) Fe & Cu in Ferric and Cupric
 - c) Fe & Mn in Ferric and manganous
 - d) Cu & Mn in Cuprous and manganous

9. How many types of wastewater treatment plants are there based on the type of wastewater?

- a) 5
- b) 4
- c) 2
- d) 3

10. On how many conditions does the intervention of wastewater depend on?

- a) 5
- b) 7
- c) 6
- d) 4

Solution: 1.c , 2.a, 3.d, 4.b, 5.a, 6.b, 7c, 8.a, 9.d, 10.b

SAMPLE VIVA QUESTION

1. List out Experiments on Water? 2. What is Reagents?
3. Explain Acidity Test?
4. Types of Acidity?
5. Explain Alkaline Test?
6. List out materials contributed towards Alkalinity?
7. How to do know about Acidity or Alkalinity of water?
8. Explain Chloride Test?
9. Effects of Chloride presence in Water?
10. Explain Total Hardness Test?
11. Hardness of water caused due the presence of which material?
12. Explain pH Test?
13. Explain Electrical Conductivity Test?
14. Explain Turbidity Test?
15. How to measure Turbidity of water?
16. What is Coagulant? Explain with Example? 17. What is the use of Chlorine?
18. How to determine Total Solids in water?
19. How to determine Total Dissolved Solids?
20. How to determine Total Fixed and Volatile Solids?
21. Explain DO Test?
22. Explain BOD Test?
23. What is the use of Flame Photometer?
24. What is need to UV Test?