

World College of Technology & Management Gurgaon

Department of Civil Engineering

Environmental Engineering Laboratory Manual



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List of Experiments:

1. Determination of Solids in Water
2. Determination of Turbidity of Water
3. Determination of Alkalinity of Water
4. Determination of Hardness of Water by EDTA Titrimetric Method
5. Determination of pH of Water
6. Determination of Chloride in Water
7. Determination of Dissolved Oxygen in Water
8. Determination of B.O.D. of Wastewater Sample
9. Determination of COD in Water
10. Determination of Odour
11. Determination of Colour

GENERAL

Instructions:

1. This laboratory manual is for reference and use for those using the environmental science/engineering laboratory.
2. Discussion after each experiment should be based on the following points:
 - (i) Limit prescribed for that constituent in drinking water standards.
 - (ii) The suitability of the sample for drinking purpose with respect to that particular constituent.
3. Students may refer the following for writing the discussion after each experiment:
 - (i) *“Manual of Standards of Quality for Drinking Water Supplies”*, Indian Council of Medical Research, New Delhi.
 - (ii) *“International Standards for Drinking Water”* — World Health Organisation.
 - (iii) “IS 2490 - 1981, IS 3306 - 1974, IS 3307 - 1977, IS 7968 - 1976, IS 2296 - 1974”, Bureau of Indian Standards, New Delhi.

DOs and DON'Ts in the Laboratory:

1. Do thoroughly clean the glassware before and after use.
2. Do handle the glassware carefully.
3. Do not handle chemicals with bare hands.
4. Do not blow out the last drop from the pipette. When the liquid has drained out completely, touch the tip of the pipette to the inner surface of the vessel.
5. Do not add water to acids. Do always add acid to water.
6. Do use large volumes of water, when a person is splashed with acid to prevent serious burns.
7. Do weigh the articles in a balance only at room temperature.
8. Do use different pipette for different reagents.
9. Do not pipette out acids and other toxic reagents by mouth.
10. Do read the level of the curve (meniscus), in all volumetric glassware, with the eye at approximately the same level as the curve of solution.

Experiment No.1 Determination of Solids in Water

Aim

The aim of the experiments is to determine the following types of solids in the given sample(s):

- (a) Total solids
- (b) Total dissolved solids
- (c) Total suspended solids
- (d) Settleable solids

Theory

'Total solids' is the term applied to the material left in the vessel after evaporation of a sample of water/waste water and its subsequent drying in an oven at a definite temperature. Total solids include "total suspended solids" the portion of total solids retained by a filter and "total dissolved solids" the portion that passes through the filter. Fixed solids is the residue remaining after ignition for 1 hour at 550°C. The solid portion that is volatilised during ignition is called volatile solids. It will be mostly organic matter. Waters that are low in organic matter and total mineral content and are intended for human consumption may be examined under 103–105°C or 179–181°C. But water containing considerable organic matter or those with pH over 9.0 should be dried at 179–181°C. In any case, the report should indicate the drying temperature.

The sample is filtered and the filtrate evaporate in a weighed dish on a steam bath, the residue left after evaporation is dried to constant weight in an oven at either 103–105°C or 179–181°C. The increase in weight over that of the empty dish represents total dissolved solids and includes all materials, liquid or solid, in solution or otherwise, which passes through the filter and not volatilised during the drying process.

The difference between the total solids and the total dissolved solids will give the total suspended solids. The dishes with the residue retained after completion of the tests for total solids and total dissolved solids are subjected to heat for 1 hour in a muffle furnace held at 550°C. The increase in weight over that of the ignited empty vessel represents *fixed solids* in each instance.

The difference between the total dissolved/total suspended solids and the corresponding fixed solids will give *volatile solids* in each instance. All the quantities should be expressed in mg/L. Settleable matter in surface and saline waters as well as domestic and industrial wastes may be determined and reported on a volume basis as millilitre per litre.

Apparatus

1. Porcelain evaporating dishes of 150–200 mL capacity
2. Steam bath
3. Drying oven
4. Desiccators
5. Weigh balance

6. Filter paper (preferably of glass fibre)
7. Electric muffle furnace
8. Imhoff cone

Procedure

(a) Total solids

1. Ignite the clean evaporating dishes in the muffle furnace for 30 minutes at 550°C and cool in a desiccator.
2. Note down the empty weight of the dish (W_1).
3. Pour a measured portion (50 to 100 mL) of the well-mixed sample into the dish and evaporate the contents by placing the dish on a steam bath.
4. Transfer the dish to an oven maintained at either 103–105°C or 179–181°C and dry it for 1 hour.
5. Allow the dish to cool briefly in air before placing it, while still warm in a desiccator to complete cooling in a dry atmosphere.
6. Weigh the dish as soon as it has completely cooled (W_2).
7. Weight of residue = $(W_2 - W_1)$ mg.
 W_2 and W_1 should be expressed in mg.

(b) Total dissolved solids

1. Filter a measured portion of the mixed sample (50 or 100 mL) through a filter paper and collect the filtrate in a previously prepared and weighed evaporating dish.
2. Repeat the steps 3 to 6 outlined in total solids procedure.
3. Weight of dissolved solids = $(W_5 - W_4)$ mg.
 W_4 = Weight of empty evaporating dish in mg.
 W_5 = Weight of empty evaporating dish in mg + Residue left after evaporating the filtrate in mg.

(c) Total suspended solids = Total solids – Total dissolved solids.

(d) Settleable solids by volume

1. Fill an imhoff cone to the litre mark with a thoroughly mixed sample.
2. Settle for 45 minutes.
3. Gently stir the sides of the cone with a rod or by spinning.
4. Settle 15 minutes longer.
5. Record the volume of Settleable matter in the cone as mL/L.

Observation:

Sr. No.	Item	Sample I	Sample II	Sample III
1	Volume of sample taken			
2	Wt. of empty evaporating dish = W_1 mg (For total solids)			
3	Wt. of dish + total solids = W_2 mg			
4	Total solids $S_1 = (W_2 - W_1)$ mg			
5	Wt. of empty evaporating dish = W_4 mg (For total dissolved solids)			
6	Wt. of dish + total dissolved solids = W_5 mg			
7	Total dissolved solids $S_2 = (W_5 - W_4)$ mg			
8	Total suspended solids in mg/L $S_3 = (S_1 - S_2)$			
9	mg/L of Settleable solids			

Calculation:

$$1. \text{ mg/L total solids} = \frac{\text{mg total solids} \times 1000}{\text{mL of sample}}$$

$$2. \text{ mg/L total dissolved solids} = \frac{\text{mg total dissolved solids} \times 1000}{\text{mL of sample}}$$

$$3. \text{ mg/L total suspended solids} = \text{mg/L of total solids} - \text{mg/L of total dissolved solids}$$

Note: These calculations need to be shown only for one sample.

Result:

Sr. No.	Item	Sample I	Sample II	Sample III
1	mg/L of total solids			
2	mg/L of total dissolved solids			
3	mg/L of total suspended solids			
4	mg/L of Settleable solids			

Discussion:

QUESTIONS:

1. What is the application of determination of Settleable solids?
2. Explain the significance of determination of total solids in sanitary engineering.
3. How will the volatile solids affect the strength of sewage? Why?
4. Why do you determine the fixed solids by igniting at 600°C? How will the result be affected, if it has Magnesium carbonate content?
5. What significant information is furnished by the determination of volatile solids?
6. What is sludge volume index?

Experiment No.2 Determination of Turbidity of Water

Aim

To determine the turbidity of the given sample using Nephelometer in NTU.

Principle

The method presented below is based on a comparison of the intensity of light scattered by the sample in specific conditions with the intensity of light scattered by standard reference suspension under the same condition. The higher the intensity of scattered lights, higher the turbidity. Formazine polymer, which has gained acceptance as the turbidity standard reference suspension is used as a reference turbidity standard suspension for water. It is easy to prepare and is more reproducible in its lights scattering properties than the clay or turbid natural water standards previously used. The turbidity of a given concentration of Formazine has an approximate turbidity of 100 NTU, when measured on candle turbidity meter. Nephelometric turbidity units based on Formazine preparation will have approximate units derived from Jackson candle turbidimeter but will not be identical to them.

Apparatus

Nephelometer with accessories

Reagents

- (i) Turbidity free distilled water (for setting zero).
- (ii) Formazine turbidity concentrate (hydrazine sulphate + hexamine).
- (iii) Formazine standard (for setting 100 of the instrument).

Preparation of Turbidity Free Distilled Water

Pass distilled water through a membrane filter having a precision pore size of less than 10 microns (Whatman filter No. 42). Rinse collecting flask at least twice with such filtered water and discard the next 200 mL. Use this filtered water for setting zero of the instrument.

Preparation of Formazine Turbidity Concentrate

(a) Solution I

Weigh accurately 5 g of 'Anal-R' quality hydrazine sulphate $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ into a 500 mL volumetric flask and add distilled water to make up to the mark. Leave the mixture to stand for 4 hours.

(b) Solution II

Weigh accurately 50g of 'Anal-R' quality hexamethylene tetramine $(\text{CH}_2)_6\text{N}_4$ (hexamine) into a 500 mL volumetric flask and add distilled water to make up to the mark.

Mix equal volume of solution I and II to form Formazine turbidity concentrate. Allow it to stand in a closed container at 25°C to 30°C for 48 hours to produce insoluble white

turbidity corresponding to 4000 NTU.

Note: Once prepared, Formazine turbidity concentrate (which corresponds to 10000 ppm SiO₂) is stable for 2 to 3 months.

Preparation of Formazine Standard

Dilute 25mL of the Formazine turbidity concentrate to 1 litre with turbidity free distilled water to obtain 250 ppm or 100 NTU for setting '100' of the instrument.

Note: Formazine standard 100 NTU should be prepared weekly.

Procedure

- (1) Switch the instrument on.
- (2) Open the lid of the sample compartment.
- (3) Insert a test tube filled with distilled water into the sample compartment. Close the lid.
- (4) Adjust 'SET 0' control to get '0' displayed on the read out.
- (5) Open the lid. Replace the test tube filled with distilled water with a test tube filled with Formazine standard. Close the lid.
- (6) Adjust the 'SET 100' control to get '100' displayed on the read out.
- (7) Repeat the above operation to get consistent values of 0 to 100 within 1% to 2%.

Measurement of turbidity less than 100 NTU

1. Thoroughly shake the sample.
2. Wait until air bubbles disappear and pour the sample into the nephelometer tube.
3. Read the turbidity directly from the instrument.

Measurement of turbidity above 100 NTU

Dilute the sample with one or more volume of turbidity free distilled water until the turbidity fall below 100 NTU.

$$\text{NTU of sample} = \frac{A(B+C)}{C}$$

Where,

A = NTU found in diluted sample B

= volume of dilution water in mL

C = sample volume taken for dilution in mL

Observation:

<i>0 – 100 NTU</i>		<i>>100 NTU</i>			
<i>Sample no.</i>	<i>NTU</i>	<i>A mL</i>	<i>B mL</i>	<i>C m L</i>	<i>NTU = A(B+C)/C</i>

Results:

<i>Description of sample</i>	<i>Turbidity in NTU</i>

Discussion:

QUESTIONS:

1. Where do you find the adverse effects of turbidity in environmental engineering? Mention two instances.
2. Discuss the significance of determination of turbidity in sanitary engineering.
3. Discuss the nature of materials causing turbidity in
 - (a) River water during flash flood
 - (b) Polluted river water
 - (c) Domestic wastewater
4. What is the standard unit of turbidity?
5. What are NTU and JTU?

Experiment No.3 Determination of Alkalinity of Water

Aim

To determine the amount of the following types of alkalinity present in the given samples:

- (a) Hydroxide alkalinity
- (b) Carbonate alkalinity
- (c) Bicarbonate alkalinity
- (d) Hydroxide–Carbonate alkalinity
- (e) Carbonate–Bicarbonate alkalinity

Principle

The alkalinity of water is a measure of its capacity to neutralize acids. It is primarily due to salts of weak acids, although weak or strong bases may also contribute. Alkalinity is usually imparted by bicarbonate, carbonate and hydroxide. It is measured volumetrically by titration with 0.02 N sulphuric acid and is reported in terms of CaCO_3 equivalent. For samples whose initial pH is above 8.3, the titration is conducted in two steps. In the first step, the titration is conducted until the pH is lowered to 8.2, the point at which phenolphthalein indicator turns from pink to colourless. This value corresponds to the points for conversion of carbonate to bicarbonate ion. The second phase of titration is conducted until the pH is lowered to 4.5, corresponds to methyl orange end point, which corresponds to the equivalence points for the conversion of bicarbonate ion to carbonic acid.

Apparatus

1. Burette
2. Erlenmeyer flask
3. Pipettes

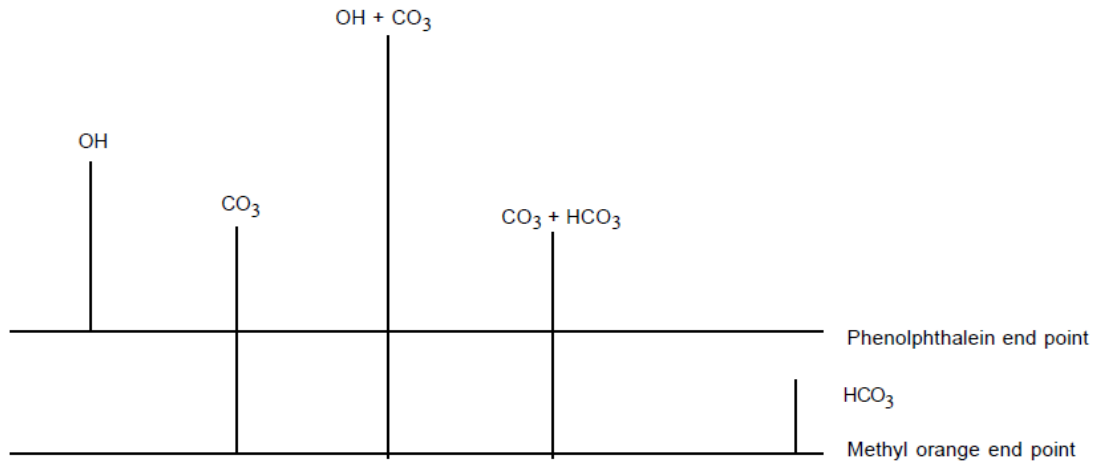
Reagents

1. Carbon dioxide free distilled water.
2. Phenolphthalein indicator.
3. Methyl orange indicator.
4. 0.1 N sodium thiosulphate solution
5. 0.02 N sulphuric acid.

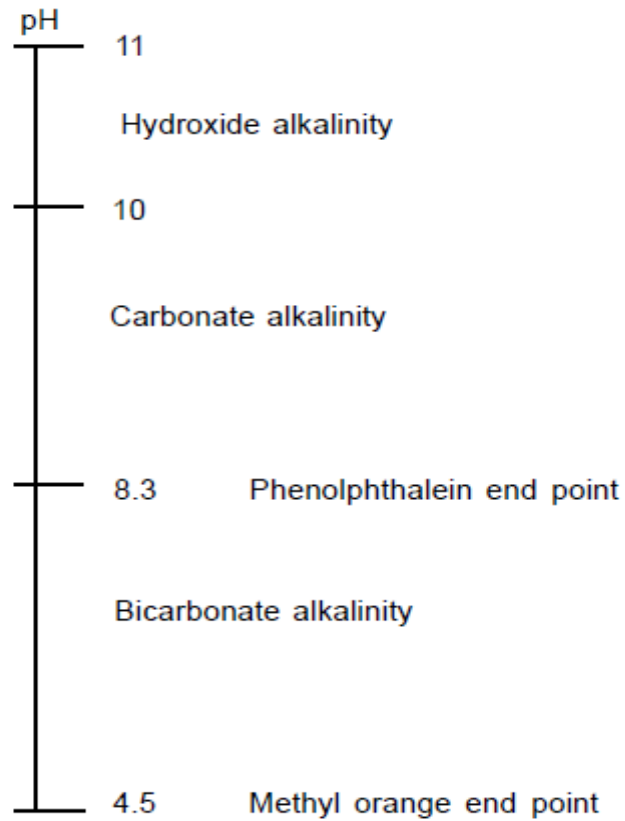
Procedure

1. Pipette 50 mL of sample into a clean Erlenmeyer flask (V).
2. Add one drop of sodium thiosulphate solution, if residual chlorine is present.
3. Add two drops of phenolphthalein indicator; if the pH is above 8.3, colour of solution becomes pink.
4. Titrate against standard sulphuric acid in the burette, till the colour just disappears. Note down the volume (V_1).
5. Then add two drops of methyl orange indicator, the colour turns yellow.

6. Again titrate against acid, until the colour turns to orange yellow. Note down the total volume (V_2).



Graphical Representation of Titration of Samples Containing Various Forms of Alkalinity



Observation

0.02 N H₂SO₄ × sample (Methyl orange/phenolphthalein indicator)

Description of sample	Trial no.	Burette reading (phenolphthalein indicator)		Volume of acid used V ₁	Burette reading (methyl orange indicator)		Volume of acid used V ₂
		Initial	Final		Initial	Final	

Calculation:

Phenolphthalein alkalinity (P) as mg/L CaCO₃ = $\frac{V_1 \times 1000}{\text{mL of Sample}}$

Total alkalinity (T) as mg/L CaCO₃ = $\frac{V_2 \times 1000}{\text{mL of Sample}}$

The types of alkalinities present in the samples are calculated using the equations given in the following table and the results are tabulated.

Result of titration	Hydroxide alkalinity as CaCO ₃	Carbonate alkalinity as CaCO ₃	Bicarbonate alkalinity as CaCO ₃
P = 0	0	0	T
P < ½T	0	2P	T – 2P
P = ½T	0	2P	0
P > ½T	2P – T	2 (T – P)	0
P = T	T	0	0

Results

<i>Descripti on of sample</i>	<i>Hydroxide alkalinity as CaCO₃ in mg/L</i>	<i>Carbonate alkalinity as CaCO₃ in mg/L</i>	<i>Bicarbonate alkalinity as CaCO₃ in mg/L</i>	<i>Hydroxide carbonate alkalinity as CaCO₃ in mg/l</i>	<i>Carbonate bicarbonate alkalinity as CaCO₃ in mg/L</i>

QUESTIONS:

1. Which is the major form of alkalinity? How is it formed?
2. What is excess alkalinity? How do you express it?
3. Why do we take 0.02 N H₂SO₄ for the titration?
4. The water where algae are flourishing is alkaline. Why? Will there be diurnal variation in pH?
5. Why does the pH change on aerating the water?
6. For efficient coagulation the water must be alkaline. Why?
7. Why do we use CO₂ free distilled water for analysis?

Experiment No.4 Determination of Hardness of Water

Aim

To determine the total hardness of the given samples by EDTA titrimetric method.

Principle

Originally, the hardness of water was understood to be a measure of the capacity of water for precipitating soap. Soap is precipitated chiefly by the calcium and magnesium ions commonly present in water, but may also be precipitated by ions of other polyvalent metals, such as aluminium, iron, manganese, strontium and zinc, and by hydrogen ions. Because, all but the first two are usually present in insignificant concentrations in natural waters, hardness is defined as a characteristic of water, which represents the total concentration of just the calcium and the magnesium ions expressed as calcium carbonate. However, if present in significant amounts, other hardness producing metallic ions should be included.

When the hardness is numerically greater than the sum of the carbonate alkalinity and the bicarbonate alkalinity, the amount of hardness, which is equivalent to the total alkalinity, is called *carbonate hardness*; the amount of hardness in excess of this is called *non-carbonate hardness*. When the hardness is numerically equal to or less than the sum of carbonate and bicarbonate alkalinity all of the hardness is carbonate hardness and there is no non-carbonate hardness. The hardness may range from zero to hundreds of milligrams per litre in terms of calcium carbonate, depending on the source and treatment to which the water has been subjected.

Ethylenediamine tetra-acetic acid and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as *Eriochrome black T* is added to an aqueous solution containing calcium and magnesium ions at a pH of 10 ± 0.1 , the solution will become wine red. If EDTA is then added as a titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex all the magnesium and calcium, the solution will turn from wine red to blue. This is the end point of the titration.

Apparatus

1. Burette
2. Pipette
3. Erlenmeyer flask
4. Bottle etc.

Reagents

1. Standard EDTA titrant (0.01 M)
2. Eriochrome black T indicator
3. Ammonia buffer solution

Procedure

1. Dilute 25 mL of sample (V) to about 50 mL with distilled water in an Erlenmeyer flask.
2. Add 1 mL of buffer solution.
3. Add two drops of indicator solution. The solution turns wine red in colour.
4. Add the standard EDTA titrant slowly with continuous stirring until the last reddish tinge disappears from the solution. The colour of the solution at the end point is blue under normal conditions.
5. Note down the volume of EDTA added (V_1).

Observation:

Sample no.	Trial No	Volume of sample (mL)	Burette reading		Volume of EDTA (mL)
			Initial	Final	

Calculation:

$$\text{Hardness as CaCO}_3 = \frac{V_1 \times S \times 1000}{V} \text{ mg/L}$$

Where, S = mg CaCO₃ equivalent to 1 mL of EDTA titrant
= 1 mg CaCO₃

$$\text{Hardness as CaCO}_3 = \frac{V_1 \times 1000}{V} \text{ mg/L}$$

Results:

Sample No. or Description	Total Hardness in mg/L as CaCO ₃

QUESTIONS:

1. What is degree of hardness? How will you classify water in terms of degree of hardness?
2. What is pseudo-hardness?
3. Explain the significance of determination of hardness of water in environmental engineering.
4. How can you remove permanent hardness from water?
5. Can you determine temporary hardness and permanent hardness separately? If yes, how?
6. What are the principal cations causing hardness in water and the major anions associated with them?
7. How is hardness classified?
8. Why is softening of water necessary? What are the advantages of soft water?

Experiment No.5 Determination of pH of Water

Aim

To determine the pH of given samples using (1) universal indicator (2) pH paper, and (3) digital pH meter.

Principle

pH value of water indicates the hydrogen ion concentration in water and concept of pH was put forward by Sorenson (1909). pH is expressed as the logarithm of the reciprocal of the hydrogen ion concentration in moles/ litre at a given temperature. The pH scale extends from 0 (very acidic) to 14 (very alkaline) with 7 corresponding to exact neutrality at 25°C. pH is used in the calculation of carbonate, bicarbonate and CO₂, corrosion and stability index etc. While the alkalinity or acidity measures the total resistance to the pH change or buffering capacity, the pH gives the hydrogen ion activity. pH can be measured calorimetrically or electrometrically. Colorimetric method is used only for rough estimation. It can be done either by using universal indicator or by using pH paper. The hydrogen electrode is the absolute standard for the measurement of pH. They range from portable battery operated units to highly precise instruments. But glass electrode is less subjected to interferences and is used in combination with a calomel reference electrode. This system is based on the fact that a change of 1 pH unit produces an electric charge of 59.1 mV at 25°C.

Apparatus

1. pH meter with electrode
2. Beaker
3. Thermometer
4. Colour comparator with discs
5. Cuvettes

Reagents

1. Buffer solutions
2. pH paper
3. Universal indicator

Procedure:

(a) Using Universal Indicator

1. 10 mL of sample is taken in a cuvette.
2. Another 10 mL sample is taken in another cuvette and 0.2 mL of universal indicator is added and placed in the hole provided for.
3. A colour disc corresponding to this indicator is inserted into the comparator and the disc rotated such that the 2 circles indicate identical colours.
4. The reading is noted.
5. The procedure can be repeated using an indicator whose range is near the value obtained.
6. The exact pH is obtained.

(b) Using pH Papers

1. Dip the pH paper in the sample.
2. Compare the colour with that of the colour given on the wrapper of the pH paper book.
3. Note down the pH of the sample along with its temperature.

(c) Using pH Meter

1. Follow the manufacturer's operating instructions.
2. Dip the electrode in the buffer solution of known pH.
3. Switch on the power supply and take the reading. Standardize the instrument using the calibrating knob.
4. After cleaning, again dip the electrodes in the buffer solution of pH 7. Note the reading. If it is 7, the instrument is calibrated. If not, correct the value and is manipulated so that the reading in the dial comes to 7.0.
5. A solution whose pH is to be found is taken in a beaker and the temperature knob is adjusted such that the temperature of solution is same as that in dial.
6. The electrode is washed with distilled water and reused with the solution and then it is dipped in the solution.
7. The reading on the dial indicates the pH of the solution.

Results

Sample no.	pH		
	pH paper	pH meter	Universal indicator
1			
2			
3			

Discussion:

QUESTIONS:

1. Discuss the relationship between (a) pH and hydrogen ion concentration (b) pH and hydroxide ion Concentration?
2. A decrease in pH of 1 unit represents how much of an increase in hydrogen ion concentration?
3. Why is it necessary to maintain the pH of water nearly 7?
4. What is a buffer solution? Give examples.

Experiment No.6 Determination of Chloride in Water

Aim

To determine the amount of chloride (in the form of Cl^-) present in the given water sample by Mohr's method.

Principle

If water containing chlorides is titrated with silver nitrate solution, chlorides are precipitated as white silver chloride. Potassium chromate is used as indicator, which supplies chromate ions. As the concentration of chloride ions approaches extinction, silver ion concentration increases to a level at which reddish brown precipitate of silver chromate is formed indicating the end point.

Apparatus

1. Burette
2. Pipettes
3. Erlenmeyer flasks
4. Measuring cylinder

Reagents

1. Chloride free distilled water.
2. Standard silver nitrate solution (0.0141N)
3. Potassium chromate indicator.
4. Acid or alkali for adjusting pH.

Procedure

1. Take 50mL of sample (V) and dilute to 100mL.
2. If the sample is coloured add 3mL of aluminium hydroxide, shake well; allow to settle, filter, wash and collect filtrate.
3. Sample is brought to pH 7–8 by adding acid or alkali as required.
4. Add 1mL of indicator (Potassium chromate).
5. Titrate the solution against standard silver nitrate solution until a reddish brown precipitate is obtained. Note down the volume (V_1).
6. Repeat the procedure for blank and note down the volume (V_2).

Observation:

Water sample vs Silver nitrate (0.0141 N) (Potassium chromate indicator)						
Sample no.	Trial no.	Volume of sample (mL)	Burette reading		Volume of silver nitrate (mL)	Chloride mg/L
			Initial	Final		
1	1					
	2					
	3					
2	1					
	2					
	3					
3	1					
	2					
	3					
Distilled Water	1					
	2					
	3					

Calculation:

For one sample (Sample No.)

V =

V1 =

V2 =

N =

$$\text{Chloride in mg/L} = \frac{(V1 - V2) \times N \times 35.46 \times 1000}{V}$$

$$= \frac{(V1 - V2) \times 500}{V}$$

$$= \quad \text{mg/L}$$

Results:

<i>Description of sample</i>	<i>Chloride concentration in mg/L</i>

Discussion:**QUESTIONS:**

1. Explain the significance of high chloride in water.
2. What are the sources of chloride in water?
3. Explain the need for blank correction.
4. Why must be the sample pH neither high nor low?
5. Why the normality of silver nitrate solution is taken as zero?
6. Would the analytical result by Mohr's method for chlorides be higher, lower, or the same as the true value if an excess indicator were accidentally added to the sample? Why?
7. What are the methods of determination of chlorides?
8. Why the water do has lower content of salt than sewage?

Experiment No.7 Determination of Dissolved Oxygen in Water

Aim

The aim of the experiment is to determine the quantity of dissolved oxygen present in the given sample(s) by using modified Winkler's (Azide modification) method.

Principle

Dissolved Oxygen (D.O.) levels in natural and wastewaters are dependent on the physical, chemical and biochemical activities prevailing in the water body. The analysis of D.O. is a key test in water pollution control activities and waste treatment process control.

Improved by various techniques and equipment and aided by instrumentation, the Winkler (or iodometric) test remains the most precise and reliable titrimetric procedure for D.O. analysis. The test is based on the addition of divalent manganese solution, followed by strong alkali to the water sample in a glass-stoppered bottle. D.O. present in the sample rapidly oxidises in equivalent amount of the dispersed divalent manganous hydroxide precipitate to hydroxides of higher valency states. In the presence of iodide ions and upon acidification, the oxidised manganese reverts to the divalent state, with the liberation of iodine equivalent to the original D.O. content in the sample. The iodine is then titrated with a standard solution of thiosulphate.

Apparatus

1. 300 mL capacity bottle with stopper
2. Burette
3. Pipettes, etc.

Reagents

1. Manganous sulphate solution ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$)
2. Alkali-iodide azide reagent
3. Conc. sulphuric acid (36 N)
4. Starch indicator
5. Standard sodium thiosulphate solution (0.025N)
6. Standard potassium dichromate solution (0.025N)

Procedure

1. Add 2 mL of manganous sulphate solution and 2 mL of alkali-iodide azide reagent to the 300 mL sample taken in the bottle, well below the surface of the liquid.
(The pipette should be dipped inside the sample while adding the above two reagents.)
2. Stopper with care to exclude air bubbles and mix by inverting the bottle at least 15 times.
3. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again.
4. After 2 minutes of settling, carefully remove the stopper, immediately add 3 mL concentrated sulphuric acid by allowing the acid to run down the neck of the bottle.

5. Re stop and mix by gentle inversion until dissolution is complete.
6. Measure out 203 mL of the solution from the bottle to an Erlenmeyer flask. As 2 mL each of manganese sulphate and azide reagent have been added, the proportionate quantity of yellow solution corresponds to 200 mL of sample is

$$= \frac{200 \times 300}{300 - 4} = 203 \text{ mL}$$

7. Titrate with 0.025 N sodium thiosulphate solution to a pale straw colour.
8. Add 1–2 mL starch solution and continue the titration to the first disappearance of the blue colour and note down the volume of sodium thiosulphate solution added (V), which gives directly the D.O. in mg/L.

Observation:

Sample x Standard sodium thiosulphate solution (0.025N) (Starch indicator)

<i>Descripti on of sample</i>	<i>Trial no.</i>	<i>Volume of sample (mL)</i>	<i>Burette reading</i>		<i>Volume of titrant mL</i>	<i>D.O. in mg/L</i>
			<i>Initial</i>	<i>Final</i>		
Sample I						
Sample II						
Sample III						

Results:

Description of Sample	D. O in mg/L
Sample 1	
Sample 2	
Sample 3	

Discussion:

QUESTIONS:

1. Discuss the environmental significance of dissolved oxygen.
2. Most of the critical conditions related to dissolved oxygen deficiency occur during summer months. Why?
3. Why do we use 0.025 N sodium thiosulphate solution for the titration?
4. The turbulence of water should be encouraged. Why?
5. Draw the oxygen saturation curve.

Experiment No.8 Determination of BOD of Waste Water Sample

Aim

To determine the amount of B.O.D. exerted by the given sample(s).

Principle

The Biochemical Oxygen Demand (B.O.D.) of sewage or of polluted water is the amount of oxygen required for the biological decomposition of dissolved organic matter to occur under aerobic condition and at the standardised time and temperature. Usually, the time is taken as 5 days and the temperature 20°C as per the global standard.

The B.O.D. test is among the most important method in sanitary analysis to determine the polluting power, or strength of sewage, industrial wastes or polluted water. It serves as a measure of the amount of clean diluting water required for the successful disposal of sewage by dilution. The test has its widest application in measuring waste loading to treatment plants and in evaluating the efficiency of such treatment systems.

The test consists in taking the given sample in suitable concentrations in dilute water in B.O.D. bottles. Two bottles are taken for each concentration and three concentrations are used for each sample. One set of bottles is incubated in a B.O.D. incubator for 5 days at 20°C; the dissolved oxygen (initial) content (D_1) in the other set of bottles will be determined immediately. At the end of 5 days, the dissolved oxygen content (D_2) in the incubated set of bottles is determined.

$$\text{Then mg/L B.O.D.} = \frac{D_1 - D_2}{P}$$

Where, P = decimal fraction of sample used.

D_1 = dissolved oxygen of diluted sample (mg/L), immediately after preparation.

D_2 = dissolved oxygen of diluted sample (mg/L), at the end of 5 days incubation.

Among the three values of B.O.D. obtained for a sample select that dilution showing the residual dissolved oxygen of at least 1 mg/L and a depletion of at least 2 mg/L. If two or more dilutions are showing the same condition then select the B.O.D. value obtained by that dilution in which the maximum dissolved oxygen depletion is obtained.

Apparatus

1. B.O.D. bottles 300mL capacity
2. B.O.D. incubator
3. Burette
4. Pipette
5. Air compressor
6. Measuring cylinder etc.

Reagents

1. Distilled water
2. Phosphate buffer solution
3. Magnesium sulphate solution
4. Calcium chloride solution
5. Ferric chloride solution
6. Acid and alkali solution
7. Seeding
8. Sodium sulphite solution
9. Reagents required for the determination of D.O.

Procedure

1. Place the desired volume of distilled water in a 5 litre flask (usually about 3 litres of distilled water will be needed for each sample).
2. Add 1mL each of phosphate buffer, magnesium sulphate solution, calcium chloride solution and ferric chloride solution for every litre of distilled water.
3. Seed the sample with 1–2 mL of settled domestic sewage.
4. Saturate the dilution water in the flask by aerating with a supply of clean compressed air for at least 30 minutes.
5. Highly alkaline or acidic samples should be neutralised to pH 7.
6. Destroy the chlorine residual in the sample by keeping the sample exposed to air for 1 to 2 hours or by adding a few mL of sodium sulphite solution.
7. Take the sample in the required concentrations. The following concentrations are suggested:

Strong industrial waste: 0.1, 0.5 and 1 per cent
Raw and settled sewage: 1.0, 2.5 and 5 per cent
Oxidised effluents: 5, 12.5 and 25 per cent
Polluted river water: 25, 50 and 100 per cent

8. Add the required quantity of sample (calculate for 650 mL dilution water the required quantity of sample for a particular concentration) into a 1000 mL measuring cylinder. Add the dilution water up to the 650mL mark.
9. Mix the contents in the measuring cylinder.
10. Add this solution into two B.O.D. bottles, one for incubation and the other for determination of initial dissolved oxygen in the mixture.
11. Prepare in the same manner for other concentrations and for all the other samples.
12. Lastly fill the dilution water alone into two B.O.D. bottles. Keep one for incubation and the other for determination of initial dissolved oxygen.
13. Place the set of bottles to be incubated in a B.O.D. incubator for 5 days at 20°C. Care

should be taken to maintain the water seal over the bottles throughout the period of incubation.

14. Determine the initial dissolved oxygen contents in the other set of bottles and note down the results.
15. Determine the dissolved oxygen content in the incubated bottles at the end of 5 days and note down the results.
16. Calculate the B.O.D. of the given sample.

Note: The procedure for determining the dissolved oxygen content is same as described in the experiment under “Determination of dissolved oxygen”.

Observation:

Sample no. or description	Concentration	Dissolved oxygen content mg/L				B.O.D. mg/L (5 days 20°C)
		Initial (D ₁)		Final (D ₂)		
		Bottle no.	D.O. value	Bottle no.	D.O. value	

Note: B.O.D. value in mg/L = $\frac{D_1 - D_2}{P}$

If concentration is 0.1 per cent, then P = (0.1/100 = 0.001) and so on

Sample calculation:

D1 = Initial Dissolved Oxygen = mg/L
 D2 = Dissolved Oxygen at the end of 5 days = mg/L
 P = Decimal fraction of sample used =

Therefore, mg/L of B.O.D. = $\frac{D_1 - D_2}{P}$ =

Results:

Description of Sample	<i>mg/L 5 days B.O.D. at 20°C</i>
Sample 1	
Sample 2	
Sample 3	

Discussion:

QUESTIONS:

1. What use is made of the B.O.D. test in water pollution control?
2. List five requirements, which must be completed with, in order to obtain reliable B.O.D. data.
3. List five requirements of satisfactory dilution water for B.O.D. test.
4. What are the three methods that can be used to control nitrification in the 5 days B.O.D. test at 20°C?
5. What are the factors affecting the rate of biochemical oxidation in the B.O.D. test?

Experiment No.9 Determination of COD of Waste Water Sample

Aim

To determine the Chemical Oxygen Demand (C.O.D.) for given sample.

Principle

Potassium dichromate is a powerful oxidising agent in acidic medium and is obtained in high state of purity. The reaction involved is:



Where, $c = 2/3n + a/6 - b/3$

C.O.D. results are reported in terms of mg of oxygen. N/8 or 0.125 N solution of oxidising agent is used in the determination. Normality double the strength is used. This allows the use of larger samples. Thus, each ml of 0.25 N solution dichromate is equivalent to 2 mg of oxygen. An excess of oxidising agent is added, the excess is determined by another reducing agent such as ferrous ammonium sulphate. An indicator ferroin is used in titrating the excess dichromate against ferrous ammonium sulphate. Blanks are used also treated and titrated to get the correct value of C.O.D.

Apparatus

1. Reflux apparatus
2. Burettes
3. Pipettes

Reagents

1. Standard potassium dichromate solution 0.25 N.
2. Sulphuric acid reagent.
3. Standard ferrous ammonium sulphate.
4. Ferroin indicator solution.
5. Mercuric sulphate.
6. Sulphuric acid crystals.

Procedure

1. Place 50.0 mL of sample in a 500 mL refluxing flask.
2. Add 1g mercuric sulphate and a few glass beads.
3. Add sulphuric acid to dissolve the mercuric sulphate and cool.
4. Add 25.0 ml 0.25 N potassium dichromate solution and mix well.
5. Attach the flask to the condenser and start the cooling water.
6. Add the remaining acid reagent (70 mL) through the open end of condenser and mix well.
7. Apply heat and reflux for 5 hours.
8. Cool and wash down the condenser with distilled water.
9. Dilute the mixture to about twice its volume and cool to room temperature.

10. Titrate the excess dichromate with standard ferrous ammonium sulphate using ferroin indicator (2 to 3 drops).
11. The colour change from blue green to reddish indicates the end point.
12. Reflux in the same manner a blank consisting of distilled water of equal volume as that of the sample.

Observation:

	<i>Burette reading</i>		<i>Volume of ferrous ammonium sulphate</i>
	<i>Initial</i>	<i>Final</i>	
Sample			
Blank			

Calculation:

$$\text{Mg/L C.O.D.} = \frac{(V1 - V2) \times N \times 8000}{V}$$

Where, V1 = mL ferrous ammonium sulphate used for blank
 V2 = mL ferrous ammonium sulphate used for sample
 N = normality of ferrous ammonium sulphate
 V = volume of sample used.

Results:

<i>Sample no.</i>	<i>C.O.D. in mg/L</i>

Discussion:

QUESTIONS:

1. Differentiate between B.O.D. and C.O.D.
2. Discuss the application of C.O.D. analysis in environmental engineering practice.
3. What are the interferences during C.O.D. test? How this can be eliminated?
4. Why ferroin is used as indicator in the C.O.D. test?
5. Why 0.25 N standard dichromate solution is used in the test?

Experiment No.10 Determination of Odour

Aim

To determine the odour of the given sample of water using human olfactory system.

Principle

The extent of odour present in a particular sample of water is measured by a term called odour intensity, which is related with the threshold odour. The threshold odour number represents the dilution ratio at which odour is hardly detectable.

Apparatus

Measuring jar, pipettes etc.

Procedure

1. Take a known volume (A) of odorous water in a measuring jar.
2. Gradually add odour free distilled water to the sample. Addition of water is continued till the sample just lost its odour. Note down the total volume.

Observation and calculation:

<i>Sample no.</i>	<i>Trial no.</i>	<i>Volume of odorous water (A mL)</i>	<i>Diluted volume (B mL)</i>	<i>Threshold odour number (B/A)</i>

Results:

<i>Sample no.</i>	<i>Threshold odour number</i>

QUESTIONS:

1. How odour can be controlled?
2. Which instrument is used for odour determination?
3. Discuss the significance of high fluorides in water supplies.
4. Discuss the significance of low fluorides in water supplies.
5. What is meant by fluoridation of water? How this can be done?
6. Explain an economical defluoridation method for drinking water supplies.
7. What are the various methods for the determination of fluoride in water?
8. Discuss the application of fluoride data.
9. What is the difference between Kjeldahl nitrogen and albuminoidal nitrogen?
10. In which form the organic nitrogen exists in domestic wastewater?

Experiment No.11 Determination of Colour

Aim

To determine the colour of given sample of water by direct observation.

Principle

The standard unit of colour is the colour produced by dissolving 1mg of platinum cobalt in 1 litre distilled water. Different standard colour intensities of solutions are prepared by dissolving known amounts of platinum cobalt with distilled water and the colour of sample is compared with the standard colour solutions.

Apparatus

Nessler tubes, volumetric flask, pipettes etc.

Procedure

1. Take 0.1mg of platinum cobalt and dissolve in 100 mL distilled water.
2. Take 1 mL, 2 mL, 3 mL etc. of this solution and dilute to 50 mL, such that we get solutions of concentrations 1/50, 2/50, 3/50, (n/50) mg/L etc. These are the comparator solutions.
3. Take the sample and the comparator solutions in the Nessler tubes and compare the colour.
4. Note down the dilution of the comparator solution having the same colour as the sample.
5. If corresponding colours are not developed, the sample may be diluted to get the corresponding colour.

Observation and calculation:

Sample no.	Dilution factor of comparator solution (n/50)	Colour unit (mg/L) (= 50/n)

Result:

<i>Sample no.</i>	<i>Colour unit in (mg/L)</i>

Discussion:

QUESTIONS:

1. Define the standard unit of colour.
2. Briefly explain the causes of colour in water.
3. Differentiate between “apparent” and “true” colour.
4. What is the reason for keeping drinking water standard for colour?
5. Why is it important to remove oil and grease from water?
6. What are the methods available to remove oil and grease from water?
7. Discuss the source and nature of acidity.
8. Discuss the significance of carbon dioxide and mineral acidity.
9. Can the pH of a water sample be calculated from a knowledge of its acidity? Why?
10. Can the carbon dioxide content of a wastewater sample known to contain significant concentrations of acetic acid be determined by the titration procedure? Why?
11. Explain why sensitive colorimetric methods are needed for the determination of nitrite nitrogen.
12. Explain the nitrogen cycle.
13. What is the significance of determination of nitrite nitrogen in water?
14. Discuss the significance of ammonia nitrogen in water.

15. What is the source of ammonia nitrogen in water?
16. In what forms does nitrogen normally occur in natural waters?
17. Discuss the significance of nitrate nitrogen analysis in water pollution control.
18. Differentiate between nitrite nitrogen and nitrate nitrogen.
19. Discuss the application of nitrate nitrogen data.
20. What are the various methods available for the determination of nitrate nitrogen?
21. What are *E. coli*? Are they harmful to human beings? Why is their presence tested in the waters to be supplied for domestic consumption?
22. What is coliform index?
23. Define MPN.
24. What are *E. coli*? Are they harmful to human beings? Why is their presence tested in the waters to be supplied for domestic consumption?
25. What is coliform index?
26. Define MPN.
27. What is disinfection? Differentiate between disinfection and sterilisation?
28. Why do we prefer chlorination over other methods of disinfection?
29. Discuss the effect of pH of water and organic matter of water on efficiency of disinfection by chlorine.
30. What is electro-katadyn process?
31. By use of appropriate equilibrium equations show why the addition of chlorine tends to decrease the pH of water, while hypochlorite tends to increase the pH.
32. Why is alum preferred to other coagulants?
33. What is the difference between coagulation and flocculation?
34. What are coagulant aids?
35. Write the significance of pH in coagulation using alum.

36. What factors affect the sedimentation of a discrete particle setting in a quiescent liquid?
37. How are the iron and manganese removed from water?
38. Explain the significance of Fe and Mn in environmental engineering.
39. In what oxidation state must the manganese be for colorimetric measurement?