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Dept. of Civil Engineering

Class:- T.Y.B Tech **Sub:-** Environmental Engineering

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Experiment no 1(A)
Determination of pH in Water sample

Aim :-

To determine the pH of given sample of water.

Theory: -

pH is defined as negative logarithm of Hydrogen ion concentration to base 10. It does express the hydrogen ion activity. It is universally used to express the intensity of the acid or alkaline condition in a solution. Since water dissociates to produce one hydroxide ion for each hydrogen ion for pure water at about 25^0 C.

$(H+)\ (OH-) = 10^{-7} \text{ X } 10^{-7} = 10^{-1}$

pH scale is usually represented as ranging from 0 to 14 with pH=7 at 25^0 C representing neutrality.

Principle:-

The **pH** electrode used in the **pH** measurement is a combined glass electrode. It consists of sensing half-cell and reference half-cell, together form an electrode system. The sensing half-cell is a thin **pH** sensitive semi permeable membrane, separating two solutions, viz., the outer solution, the sample to be analyzed and the internal solution enclosed inside the glass membrane and has a known **pH** value. An electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the **pH** of the sample.

Methodology:-

Using pH paper, pH indicator and pH meter.

Apparatus:-

1. **pH** meter
2. Beaker

Reagent:-

1. Buffers Solutions of known pH value

Sample handling and preservation :-

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage. The characteristics of the water sample may change. To reduce the change in samples taken for the determination of **pH**, keep samples at **4°C**. Do not allow the samples to freeze. Analysis should begin as soon as possible.

Precautions :-

The following precautions should be observed while performing the experiment:

1. Temperature affects the measurement of **pH** at two points. The first is caused by the change in electrode output at different temperatures. This interference can be controlled by the instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second is the change of **pH** inherent in the sample at different temperatures. This type of error is sample dependent and cannot be controlled; hence both the **pH** and temperature at the time of analysis should be noted.

2. In general, the glass electrode is not subject to solution interferences like colour, high salinity, colloidal matter, oxidants, turbidity or reductants.
3. Oil and grease, if present in the electrode layer, should be removed by gentle wiping or detergent washing, followed by rinsing with distilled water, because it could impair the electrode response.
4. Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.
5. Electrodes used in the pH meter are highly fragile, hence handle it carefully.

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.(If comparators are not available, compare the colour with colours given in the chart.)

(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

procedure of measuring pH with a pH meter is as follows:-

A) General Precautions:

1. Keep the electrode in distilled water for 24 hours before use.
2. Switch on the power supply & allow the meter to warm up for about 15 minutes.
3. Rinse the electrode with distilled water whenever it is taken out from a sample & dry it with a tissue/blotting paper before dipping the same in any other sample.
4. Keep the reading knob on standby position for all the duration when reading for pH is not being taken including rinsing period, change over period for samples etc.

B) Calibration of pH meter:

1, Keep the pH knob on pH position.
2. Keep the temperature compensation knob on Auto position.
3. Dip the electrode & temperature probe in a buffer solution of pH 4.0.
4. Keep the pH-reading knob on read position & observe its pH value.
5. if the reading is 4,0, follow the procedure from step 7 onwards,
6. If the reading is not equal to 4.0, change the reading by turning the calibration knob to read 4.0.
7. Rinse the electrode &dip it in a buffer solution of pH 9.2.
8. Keep the pH-reading knob on read position & observe its pH value.
9. If the reading is 9.2, the pH meter is ready for the measurement of pH.
10.If the reading is not 9.2, change the reading partially by turning the slope knob &

then change the reading by turning the calibration knob to read 9.2.

11. The pH meter is now ready for the measurement of pH.

C) Measurement of pH:

1. Take a sample of water in a beaker.
2. Rinse the electrode & immerse it in the sample of water,
3. Keep the pH reading knob on read position.
4. Read the pH value of sample indicated by pH meter.
5. Repeat the procedure at steps 2to 4 for the measurement of pH.

Standards for pH values :

1. The permissible value of pH in drinking water as per Bureau of Indian Standards, IS:10500 - 1983 & WHO	6.5 -8.5
2. The permissible value of pH in drinking water as per Ministry of Works & Housing, 1975, Acceptable Limit: MaximumPermissible Limit:	7.0 – 8.5 6.5 – 9.2
3. The permissible value of pH for discharge of wastewater into surface water, public sewers & for irrigation as per IS:2490 -1982	5.5 - 9.0
4. The permissible value of pH for construction water as per Bureau of Indian Standards, IS:456 - 2000, Minimum Permissible limit	6.0

Significance:

1. pH has no direct adverse effect on health. However, lower value between 4.0 produces sour taste & higher value more than 8.5 produces an alkaline taste.
2. pH can indicate the suitability of water for drinking, irrigation & construction purposes.
3, In field of water supply pH is important in chemical coagulation, disinfection, corrosion control & water softening. Each coagulant is the most effective at a given pH.
4. pH is important in the control of biological processes & it's most favourable range is 6.5 to 8.5.
5. For public water supply, pH value is 7.0 to 8.5. The lower value below 6.5 may cause corrosion of pipes whereas higher value increases the scale formation in water heating apparatus & causes incrustation, difficulty in chlorination, etc.
6. In sludge digestion process, the gaseous stage will be hindered if pH value goes below 6.0.
7. The removal of heavy metals is the maximum at a particular pH value, For example; precipitation of Chromium is maximum at a pH of 7.5.

Observation table:-

Sample No	Source of Sample	Temperature of Sample (°C)	pH

Result: -

The pH of given wastewater sample is --------

Experiment No. 1(B)

Determination of alkalinity of water

Aim: To Determine alkalinity present in the given sample of water

Apparatus: Burette, Burette stand, Measuring cylinder, conical Flask, Beaker pH meter, etc.

Reagents: pH sulphuric acid (0.02 N H2so4), Phenolphthalein Indicator, Methyl orange indicator

Theory:

Alkalinity is defined as the capacity of water to neutralize strong acids, thus, alkalinity of water is a measure of its capacity to neutralize acids. The alkalinity of natural water is primarily due to the salts of weak acids & strong bases. It is generally imparted by the salts of carbonates, bicarbonates, phosphates, nitrates, borates, etc. together with the hydroxyl ions in Free State. However, most of the waters are rich in carbonates & bicarbonates with little consideration of other alkalinity imparting ions. In general, the alkalinity of water is due to hydroxides, carbonates & bicarbonates of elements such as Ca, Mg, Na or Ammonia. Bicarbonates represent the major form of alkalinity' Most of the alkalinity in natural water is formed due to the dissolution of Coz in water, Carbonates & bicarbonates, thus formed, are dissociated to yield hydroxyl ions.

Carbonate salts produce double the hydroxyl ions than that of bicarbonates.

The alkalinity is also produced by the action of water on limestone. In the natural

& polluted water, there are many other salts of weak acids such as silicates, phosphates, borates, etc. which cause alkalinity in addition to that of carbonates & bicarbonates.

There are three forms of alkalinity viz. hydroxide, carbonate & bicarbonate alkalinity.

Principle:

Alkalinity is determined by titrating a given sample of water by standard H2SO4, first to pH 8.3 using Phenolphthalein indicator, known as phenolphthalein alkalinity (p) & then further to pH 4.5 using Methyl Orange indicator, known as Total alkalinity (T).

AII the three forms of alkalinity cannot be present at a time. Hence, either each form of alkalinity is present separately or hydroxides & carbonates or carbonates & bicarbonates

alkalinities are present. Further, it is assumed that above pH 8.3, hydroxide alkali {and half of carbonate alkalinity are present & below pH 8.3, half carbonate alkalinity & full carbonate & bicarbonate alkalinities can be calculated using the values of P & T as shown in table:

Sr. No.	Result of titration	OH Alkalinity	Cos Alkalinity	HCO_3 Alkalinity
1	P = 0	0	0	0
2	P =T	T	0	0
3	P= T/2	0	2P	0
4	P < T/2	0	2P	T-2P
5	P > T/2	2P-T	2(T-P)	0

Where,

P = Phenolphthalein alkalinity, T = Total alkalinity

Formula:

$$P\ \text{Alkalinity} = \frac{X \times N \times Weq}{\text{Volume of sample in ml}} \times 1000$$

$$\text{Total Alkalinity} = \frac{Y \times N \times Weq}{\text{Volume of sample in ml}} \times 1000.$$

Where,

X = volume of H_2SO_4 requir6d with phenolphthalein indicator in ml,

Y - Volume of H_2SO_4 required with Methyl Orange indicator in ml,

N = Normality of H_2SO_4 (0.02 N),

Weq= Equivalent weight of $CaCO_3$ in grams (50 gms).

Standard for alkalinity:

1.	The permissible value of alkalinity for $CaCO_3$ in drinking water	100 mg/lit
2.	The permissible value of alkalinity (CaCO3) for construction water as Der IS: 456-2000	250 mg/lit

Significance:

1. The alkalinity of water has little public health significance. Highly alkaline water is usually unpotable & consumers tend to seek other supplies.
2. Alkalinity in itself is not harmful to human beings, still water supplies with less than 100 mg/lit of alkalinity are desirable for domestic use.
3. The alkalinity values are important in calculating the dose of alum for water treatme4tr.
4. Alkalinity producing substances such ' corrosion in soft water supplies. It is useful in calculating lagelier saturation index (Corrosion Index).
5. Alkalinity measures are also important in controlling water treatment processes.
6. The ratio of alkalinity to that of alkaline earth metals is used to determine the suitability of irrigation water.
7. Highly alkaline waters may cause deposition of precipitates in boilers & pipes.

Bicarbonates of Ca & Mg induce temporary hardness in water, which if untreated, causes scale formation in boilers.

8. Alkalinity acts to buffer water in a pH range where the coagulation can be effective.
9. It is important in calculating lime & soda, ash requirements in softening of water.
10. It is useful in chemical coagulation, water softening, corrosion control, biological treatment & industrial waste treatment.

Procedure:

A) Phenolphthalein Alkaline:

1. Take 50 ml of sample of water in a conical flask.
2. Add 2-3 drops of phenolphthalein indicator in the conical flask containing the water sample.
3. If the colour of the sample does not become pink, it indicates P alkalinity is absent.

4. If the colour of the sample becomes pink, it indicates that P alkalinity is present & pH is greater than 8.3, then follow the procedure from step 5 to 6.

5, Titrate the sample obtained in step 4 against 0.02 N Sulphuric acid till the colour changes from pink to colourless.

6. Note down the volume of sulphuric acid required in ml (X).

B) Total Alkalinity:

1. Take 50 ml of sample of water in a conical flask.

2. Add 2-3 drops of methyl orange indicator in the conical flask containing the water sample.

3. If the colour of the sample becomes orange, it indicates that T alkalinity ipresent. Then follow the procedure from step 4 to 5.

4. Titrate the sample obtained in step 3 against 0.02 N sulphuric acid till the colour changes from orange to wine red.

5. Note down the volume of NaOH required in ml (Y).

Observation Table:

Sr. No.	Name of Water sample	Volume of Sample ml	Burette readino. ml			Alkalinity mg/l
			I.R	F. R.	Difference	

Calculation:

$$\text{P Alkalinity} = \frac{\text{X x N x Weq}}{\text{Volume of sample in ml}} \text{ x } 1000$$

=

=

$$\text{T Alkalinity} = \frac{\text{Y x N x Weq}}{\text{Volume of sample in ml}} \text{ x } 1000.$$

=

=

For P Alkalinity

1) P = x 1000

= mg/l

2) T = x 1000

= mg/l

For P Alkalinity

3) P =

4) T = x 1000

= mg/l

Now determine the relation between P & T & determine the forms of alkalinity present in the sample

Results:

The results of alkalinity measurement for the given samples are as follows:

Sr. No.	Name of Sample	Alkalinity in mg/lit, as $CaCO_3$		
		OH	CO_3	HCO_3

Conclusion:

EXPERMENT NO:-

Determination of total hardness

Aim

Determination of Total hardness of Water by EDTA method

Apparatus

Conical flask, Burette, Pipette, Beaker, Measuring cylinder

Reagents

1) 0.01M EDTA solution 2) Buffer pH 10 + 0.1 solution (NH_4CL-NH_4OH)

3) Eriochrome Black-T indicator 4) Standard hard water

5) Given water sample

Theory

Hardness is the property of water which restricts the lather formation with soap. Soaps are the sodium salts of higher fatty acids.
Hardness of water is of two types:

(I) Temporary or Carbonate hardness. It is caused by the presence of carbonates and bicarbonates of Ca2+ and Mg2+ ions. It is removed on boiling of water and sludge formation takes place.

(II) Permanent or Non-carbonate hardness. It is caused by chlorides and sulphates of calcium and magnesium and cannot be removed by boiling. For its removal water has to be treated chemically. Besides Ca2+ and Mg2+, other ions, which are causing hardness to the water, are: Fe2+, Fe3+, Mn2+, Sr2+ and AI3+. But; these ions are present in negligible amounts in the water.

Hardness of water is generally expressed as calcium carbonate equivalent, because it is the most insoluble salt and its molecular weight is 100, which makes the calculations easier.

As per degree of hardness:

Range of hardness In mg/l, as $CaCO_3$	**Classification of hardness**
0 -50	Soft
50-(75-100)-150	Moderately hard
150 - 300	Hard
>300	Very hard

Significance:

1. Hardness has no known adverse effects on health. However, it plays some role in heart diseases.
2. The presence of calcium hardness is desirable from health point of view. It causes the formation of scales in the boilers & other heat exchange equipment's like hot water pipes, solar system, etc. In boiler, carbonate & bicarbonates will release calcium corrosion.
3. The hard water is not suitable for domestic uses such as washing, cleaning & laundering.
4. Hardness is advantageous in the sense that it prevents corrosion in pipes by formatting a thin layer of scale & reduces the entry of heavy metals in the pipes to the water.
5. Hard water consume considerable amount of soap.
6. It is an important parameter for determining the suitability of water for drinking purpose & industrial use.

Procedure:

1) Take 50 ml. of water sample into a conical flask and add 3ml. of buffer solution and 3-4 drops of Eriochrome Black-T indicator.
2) Titrate this solution with standard M/100 EDTA solution taken in the burette. Titrate until the colour value is corresponds to total hardness.
3) Measure out 250 ml. of the hard water sample in 500 ml. beaker. Boil gently for half an hour; filter the solution into a 250ml. measuring flask. Make the solution up to the mark with de-ionised water and shake thoroughly. Pipette out 50ml. of this solution into a 250ml. conical flask and add 2ml of buffer solution and 3 drops of EBT indicator. Titrate with EDTA solution until the wine-red colour changes to clear blue at the point. This titre value corresponds to the permanent hardness.

Observation Table

Sr. No	Name of Water sample	Volume of Sample, ml	Burette reading, ml			Total Hardness, Mg/l
			I.R	F.R.	Diff.	

Calcualation

Part – I (Total Hardness)

1 Mole of EDTA = 1 mol. Of $CaCO_3$

1 ml. of 1 m EDTA = 100 mg of $CaCO_3$

1 ml of 0.01 m EDTA = 1 mg of $CaCO_3$

Burette reading = X ml

X ml of 0.01 m EDTA = X x 1 = X mg of $CaCO_3$

Since pipette out solution is 50ml

Hardness present in 50ml of water = X mg of $CaCO_3$

:. 1000ml of water gives (X x 1000)/50 = 40 X

Total Hardness = 40 x X mg /lit

= 40 x X ppm

Part – I (Permanent hardness)

1 ml. of 1 m EDTA = 100 mg of $CaCO_3$

1 ml of 0.01 m = 1 mg of $CaCO_3$

Burette reading = Y ml

Y ml of 0.01 m gives Y x 1 = Y

Since 50ml of water gives Y mg of $CaCO_3$

:. 1000ml of water gives (Y x 1000)/50 = 40 Y

Total Hardness = 40 x Y mg /lit

= 40 xY ppm

Temporary hardness:-

Temporary hardness = Total hardness - Permanent hardness

Precautions

1) The glass wares namely burette,pipette,beakers should be rinsed with distilled water.
2) All the solutions should be freshly prepared.
3) Distilled water should be checked before use.
4) The end point should be observed correctly.
5) pH – 10 should be maintained during the titration.

Results

(i) The total hardness of water sample is ……. ppm.
(ii) The permanent hardness of water sample is …… ppm.
(iii) The temporary hardness of water sample is …….. ppm.

Experiment No. 4 (A)

Determination of chloride content of water

Aim: To Determine the chloride content in the given sample of water.

Apparatus: Burette, Burette Stand, Measuring Cylinder, Conical Flask, Beaker, pH meter, etc.

Reagent: Silver Nitrate (0.0141 N AgNO3), Potassium Chromate indicator (K_2CrO_4) & Standard Sodium Chloride (0.01 N NaCl).

Theory:

Chlorides occur in all natural waters in widely varying concentrations. The chloride content normally increases as the mineral content increases. The most important source of chlorides in water is the discharge of domestic sewage. Man & other animals excrete very high quantities of chlorides.

About B - 15 grams of NaCl is excreted per person per day. Human excreta, particularly the urine contains chloride in an amount equal to the chlorides consumed with food & water. Therefore, sewage effluents add considerable amount of chlorides to the receiving streams. Therefore, the chloride concentration serves as an indicator of pollution of water by sewage.

It is harmless upto 150 mg/l but produces a noticeable salty taste in drinking water above 25O mg/l & hence is objectionable. It can also corrode concrete by extracting calcium in the form of calcide. Water containing MgCl produces Hydrochloric acid after heating, which makes the water corrosive & creates problems in boilers, in potable water the saline test produce by chloride concentration is variable & depends on chemical composition of water. If chloride concentrations are more than 200 mg/|, the sewage pollution is suspected. In this case bacteriological tests are recommended.

Fresh water can derive high concentration of chlorides from

1. Sewage effluents.
2. Industrial effluents.
3. Sea-water infiltration.

Principle:

Chloride content of water can be determined by the use of potassium chromate & silver nitrate. Silver nitrate reacts with chloride to form very slightly soluble white precipitate of AgCl. At the endpoint when all the chlorides get precipitated, free silver ions react with chromate to form silver chromate of reddish brown colour. If AgNO3 is added to water containing chlorides Ag++ will unite with all chloride ions forming white precipitate, before any action take place between Silver & Chromates,

Thus when red colour precipitate of Ag2CrO4 is developed in solution, at that time it is assumed that all chloride ions are precipitated. Therefore, amount of AgN03 required, to develop brick red colour in a sample to which K2CrOa has been previously added, indicates the amount of chloride present.

Formula:

$$\text{Chloride content} = \frac{\text{X x N x Weq}}{\text{Volume of sample in ml}} \text{ x } 1000 \text{ mg/l}$$

Where,

X = Volume of $AgNO_3$ in ml,

N = Normality of AgNO3 (0.0141 N),

Weq = Equivalent weight of chloride in grams (35.5 gms).

Recommended Standards for Chlorides:

1.	The permissible value of chlorides (as CI) in drinking water as per Bureau of Indian Standards, IS:10500-1983	250 mg/l
2	The permissible value of chlorides (as cl) in drinking water as per Ministry of Works & Housing, 1975 Acceptable Limit Max. Permissible Limit	 200 mg/l 1000 mg/l
3.	The permissible value of chlorides (as CI) for irrigation water	355 mg/l
4.	The permissible value of chlorides (as CI) for discharge of wastewater as per IS:2490 - 1982 Into surface water & public sewers For irrigation purpose	 1000 mg/l 600 mg/l
5.	The permissible value of chlorides (as CI) for construction water as per IS:456 – 2000 For PCC For RCC	 1000 mg/l 500 moll

Significance:

1. The chloride concentration serves as an indicator of pollution of source of water by sewage. If chloride concentrations are more than 250 mg/|, the sewage pollution is suspected. In this case, bacteriological tests are recommended.

2. It produces a noticeable salty taste in drinking water above 250 mg/l & hence, is objectionable.

3. It also corrodes concrete by extracting calcium in the form of calcide.

4. Water containing MgCl produces Hydrochloric acid after heating, which makes the water corrosive & creates problems in boilers.

5. Due to high CI concentration, water become saline, corrosion of metallic pipes & structures takes place. High chloride content in irrigation water affects the production of crops.

Procedure:

1. Take 50 ml of sample of water in a conical flask,

2. Add 2-3 drops of potassium chromate indicator in the conical flask containing the water sample. It makes the sample yellow.

3. Take silver nitrate solution in a burette.

4. Titrate the contents against AgNO3 till a permanent red precipitate is formed.

5, Note down the volume of AgNO3 required in ml (Y).

Observation Table:

Sr. No	Name of Water sample	Volume of sample, ml	Burette reading, ml			Chlorides, Mg/l
			I.R	F.R.	Diff.	

Calculation:

For sample A

$$\text{Chloride content} = \frac{\text{X x N x Weq}}{\text{Volume of sample in ml}} \text{ x } 1000 \text{ mg/l}$$

=

=

For sample B

$$\text{Chloride content} = \frac{\text{X x N x Weq}}{\text{Volume of sample in ml}} \text{ x } 1000 \text{ mg/l}$$

=

=

Results:

A) The chloride content in the given sample is …….mg/l

B) The chloride content in the given sample is …….mg/l

Conclusion:

Experiment no :- 4(B)
Residual Chlorine Test

Aim:-
Estimation of Residual Chlorine in a given sample of water.

Methodology:-
Iodometric method

Apparatus:-
1. 500 ml cap. Conical flask
2. 100 ml cap.Measuring jar
3. 25 ml pipette
4. 50 ml Burette

Reagents Used:-
1.Standard Sodium thiosulphate solution of 0.01 N.
2. Potassium Iodide (KI) crystals.
3. Glacial Acetic Acid.
4. Starch indicator Solution.

Theory:-
Since chlorine is the widely employed method for disinfection the presence of chlorine is common in potable water where chlorinated industrial effluents and sewage are discharged.The primary function of chlorination in water and wastewater is to destroy the disease causing organisms and the overall improvement of water quality. Chlorine in water may be present as free available chlorine or hypochlorite ion or both and as combined chloride. Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form combined available chlorine. Both free available chlorine and combined available chlorine liberates free Iodine with Potassium Iodide. The liberated Iodine is titrated with standard Sodium thiosulphate solution using starch as an indicator.

Principle:

Residual chlorine is determined by the starch iodide test or orthotolidine test. Chlorine is a strong oxidizing agent & liberates iodine form potassium iodide. The liberated iodine is equivalent to the amount of chlorine & can be titrated against sodium thiosulphate using starch as an indicator known as starch iodide test. This method is used when the chlorine available in water sample is more than 1.0 mg/|. In the orthotolidine test, the formation of yellow colour when orthotolidine solution is added in the sample indicates the presence of residual chlorine in the water. The intensity of this yellow colour iydompared with standard colour to determine the quantity of residual

Diagram

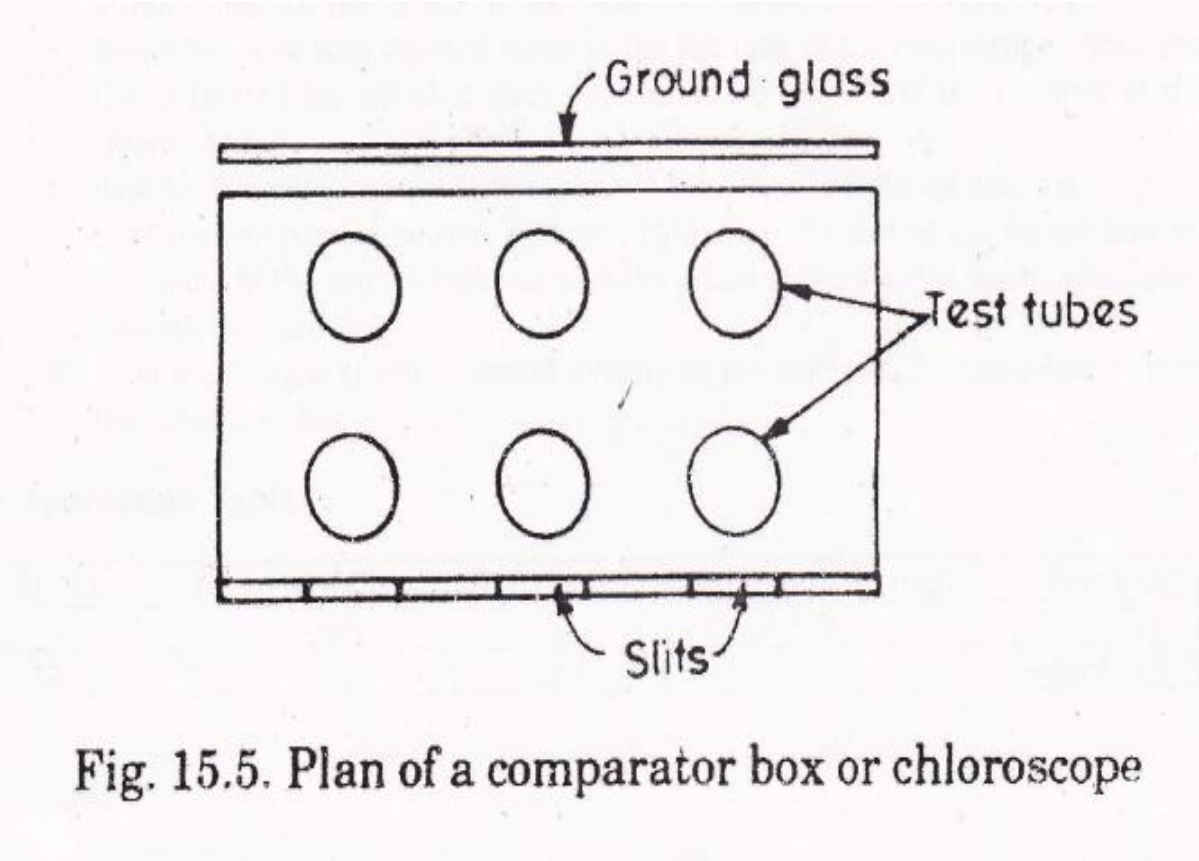


Fig. 15.5. Plan of a comparator box or chloroscope

Recommended Standards for Residual Chlorine:

1.	The permissible value for residual chlorine in drinking water as per Bureau of Indian Standards, IS:10500-1983	0.2 mg/l
2	The permissible value of residual chlorine for discharge of wastewater as per IS:2490 - 1982	1.0 mg/l

Procedure:-

1. Take 200ml of chlorinated water sample in a conical flask.
2. Add 5ml of Acetic acid and mix well. Note down the pH value, it should be 3 to 4.
3. Add 1gram of Potassium Iodide crystals and mix well.
4. Titrate immediately with Sodium thiosulphate solution (0.01 N) till light yellow color appears.
5. Add 1ml of Starch indicator, the yellow color changes to dark blue color.
6. Continue the titration till the blue color just disappears. Note down the volume of titrant used(V)

The reaction is preferably carried out in a pH of about 3 to 4

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

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

Observation:-

1. Conical Flask : 25 ml of Bleaching powder solution
2. Burette : Standard Sodium thiosulphate of 0.025N
3. Indicators : Starch solution
4. End point : Blue to colorless

Tabulation:-

Sr no.	**Sample used**	**Indicator used**	**Burette reading**			**Vol. of $Na_2S_2O_3$ used**
			FR	**IN**	**FR - IR**	
1	Bleaching powder used	Starch solution				
2						
3						

Calculations:-

mg of Chlorine present in 1mg of Bleaching powder = (Vx N x 35.45)/ ml of sample used

Therefore 1mg of bleaching powder contains X mg of Chlorine

Percentage of Chlorine in Bleaching powder =

Result:-

Experiment no 5 (A)
Determination of Turbidity of Water

Aim:

To determine the turbidity of the given sample.

Guideline:

According to WHO standard 5 NTU is suggested as the turbidity limit for drinking water, while 1 NTU is recommended to achieve the adequate disinfecting safety.

Environmental significance:

Turbidity measurements are used to determine the effectiveness of treatment produced with different chemicals and the dosages needed. Turbidity measurements help to gauge the amount of chemicals needed from day-to-day operation of water treatment works. Measurement of turbidity in settled water prior to filtration is useful in controlling chemical dosages so as to prevent excessive loading of rapid sand filters. Turbidity measurements of the filtered water are needed to check on faulty filter operation. Turbidity measurements are useful to determine the optimum dosage of coagulants to treat domestic and industrial wastewaters. Turbidity determination is used to evaluate the performance of water treatment plants.

Turbidity in water may be caused by a wide variety of suspended matter suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and other organisms. Under flood conditions, great amounts of topsoil are washed to receiving streams. As the rivers pass through urban areas, the domestic and industrial wastewaters may be added.

Principle:

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

The applicable range of this method is 0-40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample

Sample handling and preservation:

Water samples should be collected in plastic cans or glass bottles. All bottles must be cleaned thoroughly and should be rinsed with turbidity free water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal. No chemical preservation is required. Keep the samples at 4°C. Do not allow samples to freeze. Analysis should begin as soon as possible after the collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

Apparatus

Nephelometer with accessories

Standards of turbidity recommended for drinking water:

Authority	HDL	MPL
BIS	5 NTU	10 NTU
GOI	2.5 JTU	10 NTU
WHO	5 mg/l	1O mg/l

Precautions:

The following precautions should be observed while performing the experiment:

1. The presence of coloured solutes causes measured turbidity values to be low. Precipitation of dissolved constituents (for example, Fe) causes measured turbidity values to be high.
2. Light absorbing materials such as activated carbon in significant concentrations can cause low readings.
3. The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

Significance:

1. High turbidity can provide shelter for bacteria, which might be difficult to destroy during disinfection.

2. Turbidity is an important factor from aesthetic & psychological point of view.

3. Efficiency of water treatment unit is measured in terms of turbidity.

Calibration of apparatus

1. Switch on the instrument and wait till it warms up.

2. Select appropriate range depending upon expected Turbidity of the given water sample.

3. Set zero of the instrument with using distilled water (blank) and adjust zero with set zero knob.

4. Now in another test tube take standard suspension just prepared as in selection for 0 to 200 NTU range, use 100 NTU solution.

5. Set display to the value of standard Suspension with calibration knob.

6. Now the instrument is ready to take measurement of any solution of unknown turbidity.

Procedure

1. The Nephelometer turbidimeter in switched on and waited for few minutes till it warms up.
2. The instrument is set up with a 4ONTU standard suspension
3. The sample is thoroughly shaked and kept it for sometimes so the air bubbles are eliminated
4. The sample is taken in Nephelometer sample tube and the sample is put in Sample chamber and the reading is noted directly.
5. The sample is diluted with turbidity free water and again the turbidity is read.

Observation Table

Sample No	Source of Sample	Temperature of Sample (°C)	Turbidity (NTU)

Result:-

The turbidity for given water sample is = ----------

Experiment No. 5 (B)

Determination of optimum dose of alum

Aim: To Determine the optimum dose of alum by jar test.

Apparatus: Jar test apparatus with at least of adjustable speed.

Reagents: Alum solution (1%)

Lime solution as $Ca(OH)_2$ (1%)

Theory:

The most conventional of removal of raw water turbidity, caused by colloidal & fine suspended impurities in the particle size range of 10-7 to 10-3 cm, is by coagulation & flocculation, followed by clarification & filtration.

Coagulation is neutralization of negative charges on colloidal impurities. (The repulsive zeta potential forces are reduced to an extent, that intermolecular attractive forces become predominant in the system.) Coagulation is carried out by quick or flash mixing with coagulants, generally based on aluminium & to a less extent on iron, which yield positively charge metal ions on dissociation & floe forming metallic hydroxides on hydrolysis. Flash mixing is brought about by rapid mechanical stirring or turbulent hydraulic agitation.

Flocculation is coalescence of coagulated particles to form larger agglomerates which consist of turbid particles (comprising silt, clay, algae, amoebic cysts, Bacteria, viruses, etc). and alum floe with entrained water. Flocculation is brought about mechanically by slowly rotating paddles so controlled as not to break up formed floe masses.

The jar test is a laboratory test, which comprises the three unit operations, coagulation, flocculation & clarification.

The purpose of the test to determine the optimum dose i.e. the lowest coagulant dose which gives maximum clarification so that the residual turbidity will be in the range of 5 - 10 NTU, a suitable load on filters.

Procedure:

1. Take 500 ml of well mixed raw water in each of four one litre jars A, B, C & D. Measure its turbidity with the help of turbidity meter.

Shake well & add graded dosages of \o/o alum solution to each jar. For example add 20, 30, 40 & 50 mg/l dose of alum to jar A, B, C & D respectively. (i.e. add 1.0, 1.5, 2.0 & 2,5 ml/500 ml dose of alum to jar A, B, C & D respectively.

3. Flash mix. i.e. stir the contents vigorously for one minute.

4. Flocculate, by reducing the speed to be in the range of 20 - 40 rpm, for 20 for 20 minutes,

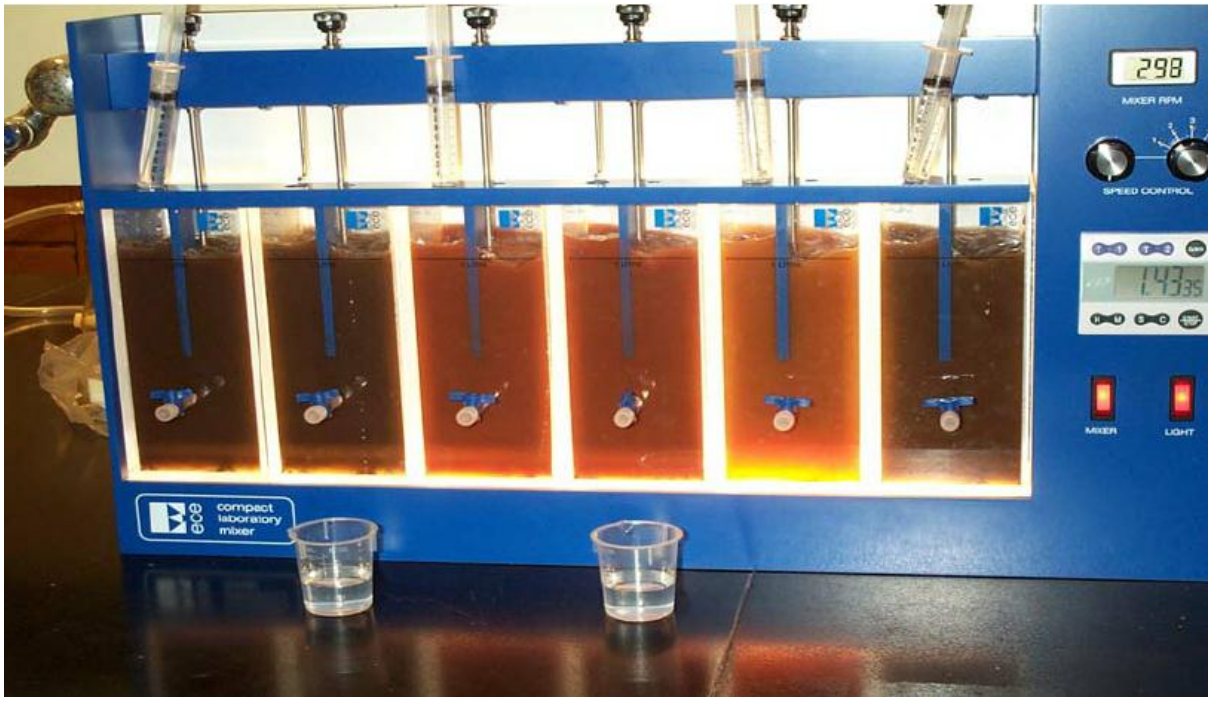
5.Stop the rotation of paddles. Take out the jars & allow them to settle for 20 minutes.

6. Draw the supernatant from each jar.

7. Plot a graph of residual turbidity (mg/l) along Y-axis against alum dosage (mgll) along X-axis.

8. Determine the optimum dose of alum corresponding to a residual turbidity of 10 mg/l (In some treatment plants a residual turbidity of 5 mg/|, after chemically assisted sedimentation, may be specified as a suitable load on filters.)

Diagram



Observation Table

Jar	**Raw water Sample**	**Dosage of 1olo alum**		**Dosage of lo/o $Ca(OH)_2$**		**Turbidity mg/l**
		mg/l	ml/500 ml	mg/l	ml/500 ml	

Results:

Optimum dose of alum to be adopted, for coagulation in the water treatment plant is

……..mg/l

Conclusion:

Experiment No. 6

Determination of MPN

Aim : To introduce concepts of total coliforms using the MULTIPLE-TUBE FERMENTATION TECHNIQUE

Background:

Read Handout Standard Methods 9221 MULTIPLE-TUBE FERMENTATION TECHNIQUE FOR MEMBERS OF THE COLIFORM GROUP (section 9221A to 9221C). In summary, coliforms group of bacteria ferment lactose and produce gas. a broth containing lactose and other substances which inhibit noncoliform organisms is placed in series of test tubers which are then inoculated with a decimal fraction of 1 mL(100,10,1,0.1,0.01, etc.). These tubes are incubated at the appropriate temperature and inspected for development of gas. The first stage is called the presumptive test and tubes with gas developed are presumed to have coliforms present (we will do till this stage). A similar is test, called as confirmed test, is set up to confirm the presence of coliforms organisms. See following schematic of all test involved.

A statistical method in conjunction of following table is used to determine the most probable number of coliform bacteria in 100 mL of sample. When more than 3 dilutions are used in decimal series of dilution, select the three most appropriate dilutions refer following table.

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 10 ml	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

TABLE 9221.IV. MPN INDEX AND 95% CONFIDENCE LIMITS FOR VARIOUS COMBINATIONS OF POSITIVE RESULTS WHEN FIVE TUBES ARE USED PER DILUTION (10 mL, 1.0 mL, 0.1 mL)

Combination of Positives	MPN Index/ 100 mL	95% Confidence Limits		Combination of Positives	MPN Index/ 100 mL	95% Confidence Limits	
		Lower	Upper			Lower	Upper
0-0-0	<2	—	—	4-2-0	22	9.0	56
0-0-1	2	1.0	10	4-2-1	26	12	65
0-1-0	2	1.0	10	4-3-0	27	12	67
0-2-0	4	1.0	13	4-3-1	33	15	77
				4-4-0	34	16	80
1-0-0	2	1.0	11	5-0-0	23	9.0	86
1-0-1	4	1.0	15	5-0-1	30	10	110
1-1-0	4	1.0	15	5-0-2	40	20	140
1-1-1	6	2.0	18	5-1-0	30	10	120
1-2-0	6	2.0	18	5-1-1	50	20	150
				5-1-2	60	30	180
2-0-0	4	1.0	17	5-2-0	50	20	170
2-0-1	7	2.0	20	5-2-1	70	30	210
2-1-0	7	2.0	21	5-2-2	90	40	250
2-1-1	9	3.0	24	5-3-0	80	30	250
2-2-0	9	3.0	25	5-3-1	110	40	300
2-3-0	12	5.0	29	5-3-2	140	60	360
3-0-0	8	3.0	24	5-3-3	170	80	410
3-0-1	11	4.0	29	5-4-0	130	50	390
3-1-0	11	4.0	29	5-4-1	170	70	480
3-1-1	14	6.0	35	5-4-2	220	100	580
3-2-0	14	6.0	35	5-4-3	280	120	690
3-2-1	17	7.0	40	5-4-4	350	160	820
4-0-0	13	5.0	38	5-5-0	240	100	940
4-0-1	17	7.0	45	5-5-1	300	100	1300
4-1-0	17	7.0	46	5-5-2	500	200	2000
4-1-1	21	9.0	55	5-5-3	900	300	2900
4-1-2	26	12	63	5-5-4	1600	600	5300
				5-5-5	≥1600	—	—

When the series of decimal dilutions is different from that in above table, select the MPN value from above table

and calculate according following formula:

MPN/100 mL = (Table MPN/100 mL)*(10/V)

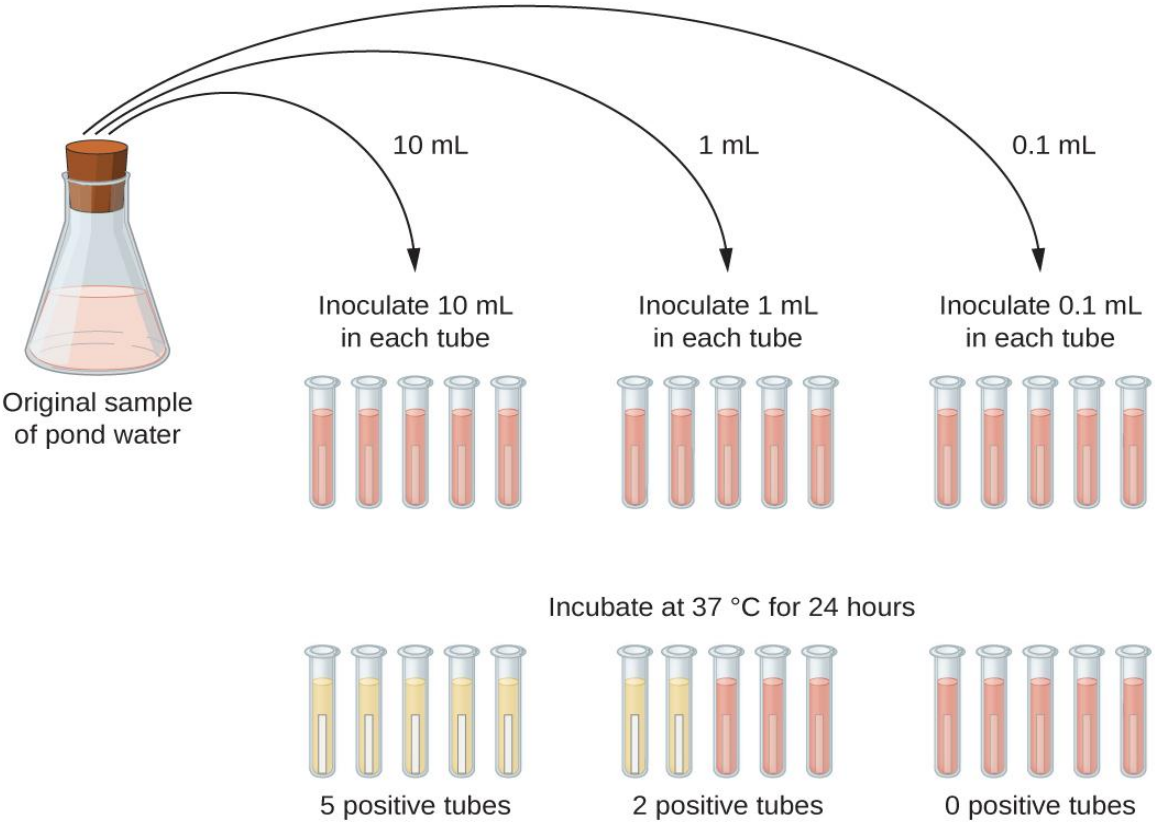
Where V=volume of one sample portion at the lowest selected dilution

Example calculation: Determine MPN of coliform organisms

Example	**Sample volume (ml)**					**Combination Of positives selected**	**MPN index No./100ml**
	10	1	0.1	0.01	0.001		
No. positive	4	2	1	1	0		
No. negative	1	3	4	4	5		

Select a series where tubes each have positive results. Use sample size 10, 1, 0.1 ml (with combination of positives: 4-2-1).

From table, MPN/100 mL comes out to be 26 (range: 9-78 organisms/100 mL possible at 95% confidence level).



Experiment no. 7

SULPHATE CONTENT

Aim: - To determine the sulphate content of given sample.

Apparatus: - * Nephelometric turbidity meter with sample cells.

* Magnetic stirrer.

* Timer with indication of second.

Reagent: - * Buffer sol^{n} A

* Buffer sol^{n} B

*Barium chloride, Bacl2, Crystal 20 to 30

meal. Standard sulphate sol^{n} Stand.

Sodium carbonate.

*std. $H2SO_4$

Nomraiity N= A*B/53.00 xC

*std. sulphuric acid

Procedure:-

1) Standard Nephelometric following manufacture instruction.
2) Measure the turbidity of sample blank. A sample in witch nos bacl2 is added.
3) Measure 100ml sample.
4) Measure turbidity of the sample of 5 0.5min after stirring ended.
5) Prepare so4^2 stand. At 5mg/L

SOa2- according to the following protocol.

So4^2 mg/ L					
std. so4^2 Sol^n mI					
Distilled water rnl					

6) Develop base turbidity for the std. as above
7) ln case of buffer solution B is used for sample containing less than l0 mg/L SO4 run a reagent blank with distilled water in place of sample developing turbidity and reading a above.

Calculation: - in case buffer solution A is used read so4 concentration for the sample from the calibration curve after substation the turbidity of the treated sample. If less than 100ml sample was used multiple the result by l00ml sample volume"

Experiment no- 08(A)

Determination of fluoride content

Aim

To determine the fluoride content in water.

Principle

Fluorides in excessive quantities and absence of fluorides in water, both create problems. A disfigurement in teeth of humans known as mottled enamel or dental fluorosis is occured in those, who consume waters with fluoride content in excess of 1.0 mg/L. It has been scientifically established that 0.8-1.0 mg/L of fluorides is essential in potable water. Thus, absence or low fluoride content may cause dental caries in the consumers.

Fluorides are measured by colorimetric methods. Fluorides are separated out by distillation, if interfering substances are present. Fluorides are analysed by a method that involves the bleaching of a performed colour by the fluoride ion. The performed colour is the result of the action between zirconium ion and alizarin dye. The colour produced is referred to a lake and the intensity of colour produced is reduced if the amount of zirconium present is decreased. Fluoride ion combines with zirconium ion to form a stable complex ion ZrF_6^{--} , and the intensity of the colour lake decreases accordingly. The reaction is as follows:

Zr_alizarin lake + 6F	→	alizarin + ZrF_6^{--}
(reddish colour)		(yellow)

The bleaching action is the function of the fluoride ion concentration and is directly proportional to it. Thus, Beer's law is satisfied in and inverse manner.

Apparatus

1. Spectrophotometer or colour comparator

Reagents

Standard fluorides solution 1mL = 10µgF.

1. Zirconyl-alizarin reagent.
2. Mixed acid solution.
3. Acid-zirconyl-alizarin reagent.
4. Sodium arsenite solution.

Procedure

If residual chlorine is present, remove the same by adding one drop of arsenite per 0.1 mg Cl and mix.

Prepare a series of standard by diluting various volume of standard fluoride solution (1 ml =10 µgf) to 100 mL in tubes. The range should be such that it is between 0 and 1.4 mg/L.

To 50 mL of each standard add 10 mL mixed acid-zirconyl-alizarin reagent.

Set the spectrophotometer to a wavelength of 570 nm.

Adjust the spectrophotometer to zero absorbance with the reference solution i.e., distilled

water with reagent.
Plot the concentration along x-axis and absorbance along y-axis and obtain a calibration curve.
Take 50 mL of the sample and add 10 mL of mixed acid-zirconyl-alizarin reagent and mix well.
Place the solution in the spectrophotometer and read the absorbance.
By referring the calibration curve, the concentration for the observed absorbance is read out.
Repeat the procedure with dilute samples.

Observation

The observation is presented in Tables A and B respectively.

Table A: Observation for calibration

Stock fluoride solution in ml	Fluoride	Absorbance

Table B:

Sample no	Absorbance	Fluoride in µg from graph	Fluoride in mg

Calculation

F in mg/L = $\frac{(A \times B)}{(V \times C)}$

where,

A = μgF determined

B = sample dilute to this volume

C = portion taken for colour development

V = mL of sample.

Results

Sample no. or description	**Fluoride in mg/l**

Experiment no- 08 (B)

Determination of iron content

Aim

To determine the iron content of an unknown sample.

Summary

Iron +II is reacted with o-phenanthroline to form a coloured complex ion. The intensity of the coloured species is measured using a Spectronic 301 spectrophotometer. A calibration curve (absorbance versus concentration) is constructed for iron +II and the concentration of the unknown iron sample is determined.

Theory

Colorimetric analysis is based on the change in the intensity of the colour of a solution with variations in concentration. Colorimetric methods represent the simplest form of absorption analysis. The human eye is used to compare the colour of the sample solution with a set of standards until a match is found.

An increase in sensitivity and accuracy results when a spectrophotometer is used to measure the colour intensity. Basically, it measures the fraction of an incident beam of light which is transmitted by a sample at a particular wavelength. You will use a Spectronic 21 in this experiment.

Safety

The wearing of safety glasses/goggles is mandatory at all times. Those students wearing prescription glasses must wear goggles over their glasses. Students without prescription lenses

must wear the safety glasses provided. Contact lenses should not be worn in the lab. Safety glasses/goggles

Procedure: (note - work in pairs)

1. The standard iron solution contains 0.25g/l of pure iron. Pipet 25.00 ml of this standard iron solution in 500ml volumetric flask & dilute upto the mark with distilled water.
2. Prepare the following iron calibration solutions by pipetting the indicated amounts of the above iron solution (step 1) into labeled 50 mL volumetric flasks. The first flask is a blank containing no iron.

Concentration of Fe	Volume of pipet
0.00 mg Fe	0.00 mL
0.05 mg Fe	4.00 mL
0.10 mg Fe	8.00 mL
0.15 mg Fe	12.00 mL
0.20 mg Fe	16.00 mL
0.25 mg Fe	20.00 mL

3. Pipet 10.00 mL of an unknown sample solution (record the unknown's number) into a 250 mL volumetric flask and dilute to the mark with distilled water. Invert and shake the flask several times to mix the solution.
4. Pipet two 25.00 mL aliquots of this solution into two 50 mL volumetric flasks labeled unknown.
5. Using a 10 mL graduated cylinder, add 4.0 mL of 10% hydroxylamine hydrochloride solution and 4.0 mL of 0.3% o-phenanthroline solution to each volumetric flask.

6. Swirl and allow the mixture to stand for 10 minutes.
7. Dilute each flask to the mark with distilled water and mix well by inverting and shaking
the capped volumetric flasks several times.
8. Using the Spectronic 301 spectrophotometer, carefully measure the percent transmittance
of the various solutions in the 50 mL volumetric flasks, including the two unknown solutions. Record your results in the following table.

1.

Solution	% Transmittance	Absorbance (A=-logT)
0.00 mg Fe (blank)	100%	0
0.05 mg Fe		
0.10 mg Fe		
0.15 mg Fe		
0.20 mg Fe		
0.25 mg Fe		

Calculations and Discussion

1. Prepare a plot of absorbance versus concentration of the known solutions (express the concentration in mg Fe per 50 mL of solution). Draw the best fitting straight line through the points – this is called the **Beer-Lambert Law**plot.
2. Place the best Absorbance value of each unknown solution onto this plot and determine their concentrations.
3. Calculate the amount of iron in the unknown sample. Express this as mg of Fe per litre of the original unknown solution (mg/L Fe).
E.g. From the graph you obtain a concentration of 0.10 mg Fe/50 mL
Since in step 3 we diluted the original sample 25 times and in step 4, 2 more times the concentration of the original sample is therefore:

Unknown #1 173.5 mg/L	Unknown #2 209.2 mg/L	Unknown #3 225.6 mg/L	Unknown #4 242.7 mg/L

0.10mg Fe/ 50mL x 50 (dilution factor) x 1000ml /L + 100mg Fe/ L

And calculate relative error

relative error = (experimental value + accepted value) / accepted value x 100

Experiment No. 09

Determination of total solid in water

Aim: To determine the different types of solids present in the given sample of water.

Apparatus: Balance, weight box, Porcelain dish, Silica crucible, Filter paper, Beakers, Funnel with its holder, Measuring cylinder, oven, Desiccators, etc.

Theory:

The term solids refers to the matter that remains as residue upon Evaporation & drying at 103 - 105 0c in an oven. This is called as Total solids. This includes suspended as well as dissolved solids, volatile as well as fixed solids & organic as well as inorganic solids. To determine volatile solids, the ignition of the sample should be carried at 650 ^{0}C in a muffle furnace. Use of porcelain dishes for total solid determination should avoided be because of their tendency to change weight & therefore, platinum dishes are mostly used.

There are five forms of solids viz. total solids, suspended solids, dissolved solids, volatile solids & fixed solids. Dissolved solids, also referred to as total dissolved solids, indicate mainly the various kinds of minerals present in the water. However, if some organic substances are also present, as in the case of the polluted waters, they may also contribute to the dissolved solids. In natural waters, dissolved solids are composed of carbonates, bicarbonates, chlorides, sulphates, phosphates & nitrates of Ca, Mg, K, Fe, Mn, etc.

Further, solids in sewage consist of inorganic as well as organic solids. The organic solids are about 45o/o of total solids & the inorganic solids are about 55 o/o of total solids' The total suspended solids in sewage may contain volatile as well as fixed matter' Fixed solids are determined as the residue left after ignition of the total suspended solids at 550 0c in a furnace for about 15 - 20 minutes. The volatile solids are determined as the difference between the total suspended solids &the fixed solids.

Principle:

Total solids are determined as the residue left after evaporation & drying of the unfiltered sample at 103 - 105 ^{0}c in an oven. Total dissolved solids are determined as the residue left after evaporation & drying of the filtered sample at 103 -105 0C in oven. Total

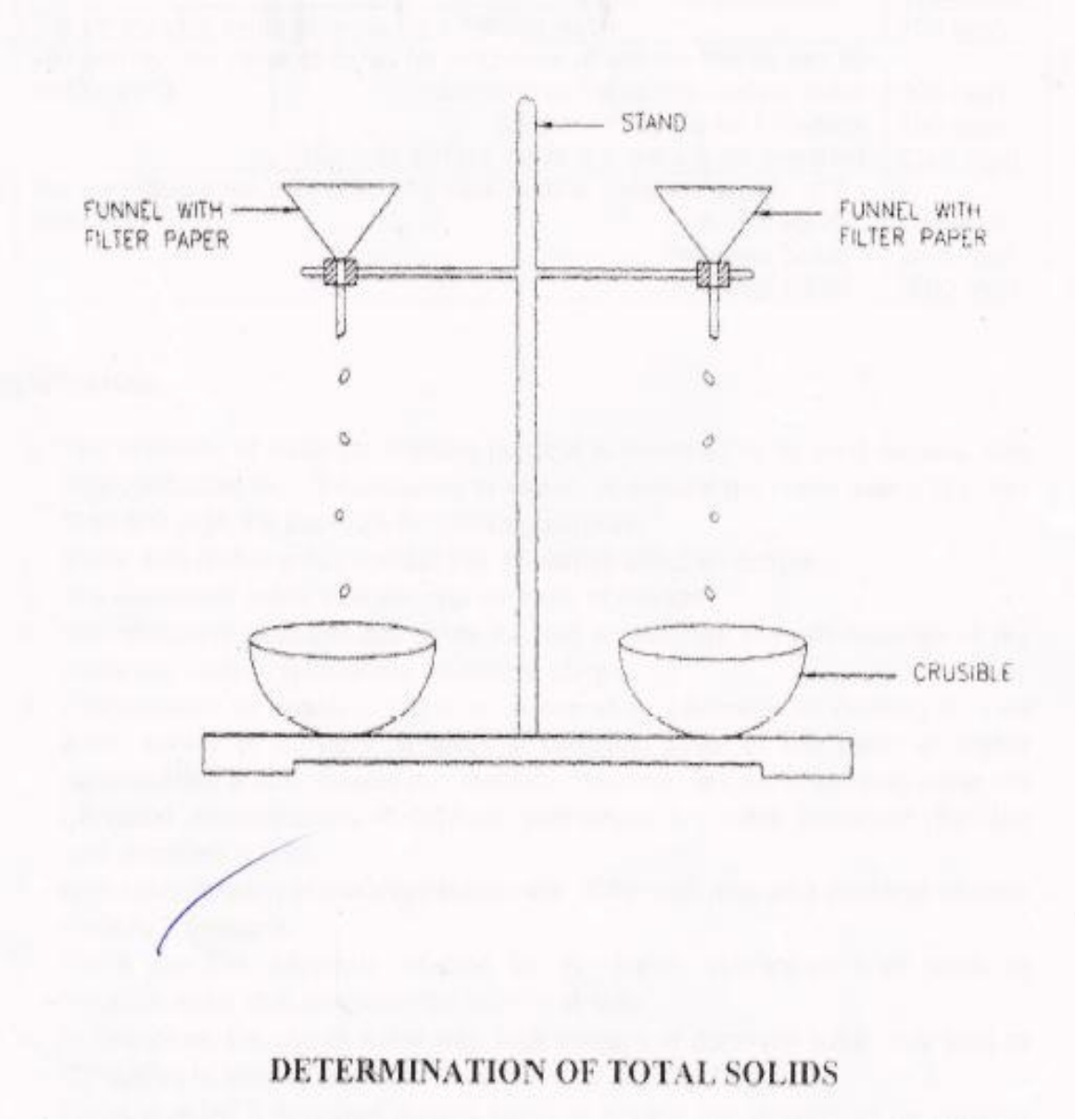
suspended solids are determined as the difference between total solids & the total dissolved solids.

$$\text{Total solid} = \frac{(wz - w)x\ tooo\ x\ tooo}{\text{Volume of sample in ml}} \quad \text{mg/l}$$

$$\text{Total dissolved solid} = \frac{(w4 - w3)x\ tooo\ x\ tooo}{\text{Volume of sample in ml}} \quad \text{mg/l}$$

Total Suspended Solids =

Diagram



Total Solids - Total Dissolved Solids mg/l

Where,

W_1 = Weight of empty dish in grams,

W_2 = Weight of dish plus residue for unfiltered sample in grams,

W_3 = Weight of empty dish in grams,

W_4 = Weight of dish plus residue after drying filtered sample in grams.

Recommended Standards for solids:

1.	The permissible value of total dissolved solids Ministry of Works & Housing,1975 in drinking water as per Acceptable Limit Max. Permissible Limit	 500 mg/l 1500 mg/l
2.	The permissible value of solids for irrigation water	700 mg/l
3.	The permissible value of solids for discharge of wastewater as per IS: 2490 – 1982 Suspended Solids into surface water Suspended Solids for irrigation TDS into surface water & sewers & for irrigation	 100 mg/l 200 mg/l 2100 mg/l
4.	The permissible value of solids for construction water as per IS: 455 2000 Organic Solids Inorganic Solids Suspended Solids	 200 mg/l 3000 mg/l 2000 mg/l

Significance:

1. The suitability of water for drinking purpose is governed by its solid content. High solids indicates the contamination of water. In general the water with a TDS less than 500 mg/l are desirable for drinking purpose.

2. Water with higher solids content has a laxative effect on people.

3. The suspended solids indicates the strength of sewage,

4. Determination of suspended solids is used to measure the effectiveness of the treatment units in wastewater treatment plants.

5. Concentration of dissolved solids is an important parameter in drinking & other water quality parameters. It gives a particular taste to the water at higher concentration & also reduces its portability. However, in case of drinking water the individual concentrations of different substances are more important than the total dissolved solids.

6. High concentration of dissolved solids near 3000 mg/l may also produces distress in cattle & livestock.

7. Plantsjlval.so adversely affected by the higher concentration of solids in irrigation water that increases the salinity of soils.

8. In industries, the use of water with high amount of dissolved solids may lead to the scaling in boilers, corrosion & degrade the quality of product.

9. The suspended & dissolved organic solids in sewage are responsible for creating nuisance, if dispersed without treatment.

10. Suspended solids are important in the design of settling tank.

Procedure:

A) Total Solids:

1. Take a porcelain dish & find its empty weight, W_1.

2. Take 100 ml sample of unfiltered water in the dish.

3. Dry the sample in an oven at 103 -105 ^{0}C for about 24 hours.

4. Find the final weight of the dish including the residue, W_2.

B) Total Dissolved Solids:

1. Take a porcelain dish & find its empty weight, W_3,

2. Filter 100 ml sample of water using filter paper No. 41

3. Take the filtered sample of water in the dish.

4. Dry the sample in an oven at 103 -105 ^{0}C for about 24 hours.

5. Find the final weight of the dish including the residue, W_4.

Observation Table:

Sr. No	Name of Water sample	Type of solid	Weight of dish, gram			Solids, Mg/l
			I.R	F.R.	Diff.	
		TS				
		TDS				

Calculation:

$$\text{Total solid} = \frac{(wz - w)x\ tooo\ x\ tooo}{\text{Volume of sample in ml}} \quad \text{mg/l}$$

=

=

$$\text{Total dissolved solid} = \frac{(w4 - w3)x\ tooo\ x\ tooo}{\text{Volume of sample in ml}} \quad \text{mg/l}$$

=

=

Result:

The results of solids measurement for the given sample are as follows:

Sr. No	Name of sample	Solids in mg/l		
		TS	TDS	SS

Conclusion:

EXPERIMENT NO: 10

SLUDGE VOLUME INDEX (SVI)

AIM: To determine SVI of the provided sample.

PRINCIPLE: SVI determination is based on estimating the volume of sludge settled in 30 minutes per gram of MLSS (mixed liquor suspended solids).

APPARATUS: Imhoff cone or 1000ml measuring cylinder, 50 ml measuring cylinder, crucibles, beaker, Whatman filter paper no.40, funnel, stand, hot air oven.

PROCEDURE:

1. Take exactly one liter of a mixed liquor sample and allow it to settle in on Imhoff cone.

2. Record the volume of settled sludge (V ml/l) at the end of 30 minutes.

3. Weigh a clear and empty crucible as (W1)

4. Stir up the Imhoff cone contents well, collect 50ml of the mixed liquor in a crucible and evaporate to dryness in a hot air oven.

5. Cool the crucible to room temperature and weigh with solids residue as (W2)

6. Weigh another clean and empty crucible as (W3)

7. Stir up the Imhoff cone content again and collect 50ml of the mixed liquor and filter through Whatman filter paper no.40 and collect the filtrate in the crucible and evaporate to dryness.

8. Cool the crucible with dissolved solids residue and weigh as (W4).

OBSERVATIONS:

1. Weigh of empty crucible (W1)= _____ gm

2. Weigh of crucible with unfiltered residue (W2) = _____gm

3. Weigh of another empty crucible (W3) = _____gm

4. Weigh of crucible with filtered residue (W4)= _____gm

5. Volume of settled sludge (V) = _____ _ml

CALCULATIONS:

1. Mixed liquor total solids (MLTS), mg/l = (W2-W1) x 1000 Sample taken

2. Mixed liquor dissolved solids (MLDS), mg/l = (W4-W3) x 1000 Original sample taken

3. Mixed liquor suspended solids (MLSS), mg/l = MLTS – MLDS SVI ml/g = V x1000 MLSS

RESULT: The amount of SVI determined from the provided sample is _____

ENVIRONMENTAL SIGNIFICANCE:

1. The value of SVI is of operational importance since it reflects changes in the treatment system.

ANALYSIS OF DATA:

1. SVI is used for determining the quality of sludge produced in a biological aeration unit and hence its efficiency.

2. It is used for determining the recirculation ratio necessary for maintaining a specified MLSS concentration in the aerator.

3. It is also used for estimating suspended solids concentration in recirculated sludge.

CONCLUSION:- The sludge volume index of the given sample is _____ .

Experiment No. 11

Determination of dissolved oxygen in water

Aim: To Determine the concentration of oxygen demand in the given sample of water

Apparatus: Burette, Burette Stand, Measuring Cylinder, Conical Flask, Beaker, BOD bottle with stopper, graduated pipette, etc.

Reagents:

1. Manganese Sulphate solution,
2. Potassium Iodine solution,
3. Standard Sodium Thiosulphate solution,
4. Concentrated Sulphuric Acid,
5. Starch Indicator.

Theory:

Dissolved Oxygen (DO) level depends on physical & biochemical activities prevailing in water body. There are two methods for analysis iodometric & electrometric. The iodometric method is based on the oxidizing property of DO while the membrane electrode procedure is based on the rate of diffusion of molecular oxygen across a membrane.

In wastewater, dissolved oxygen is factor that determines whether a hydrological process undergoing a change or aerobic low values of dissolved oxygen adversely affects the portability of the water & may cause killing of aquatic life.

Temp. ^{0}C	0	5	10	15	20	25	30	35	40	45
D.O. mg/l	14.6	12.8	11.2	10.2	9.2	8.4	7.6	7.1	6.6	6.1

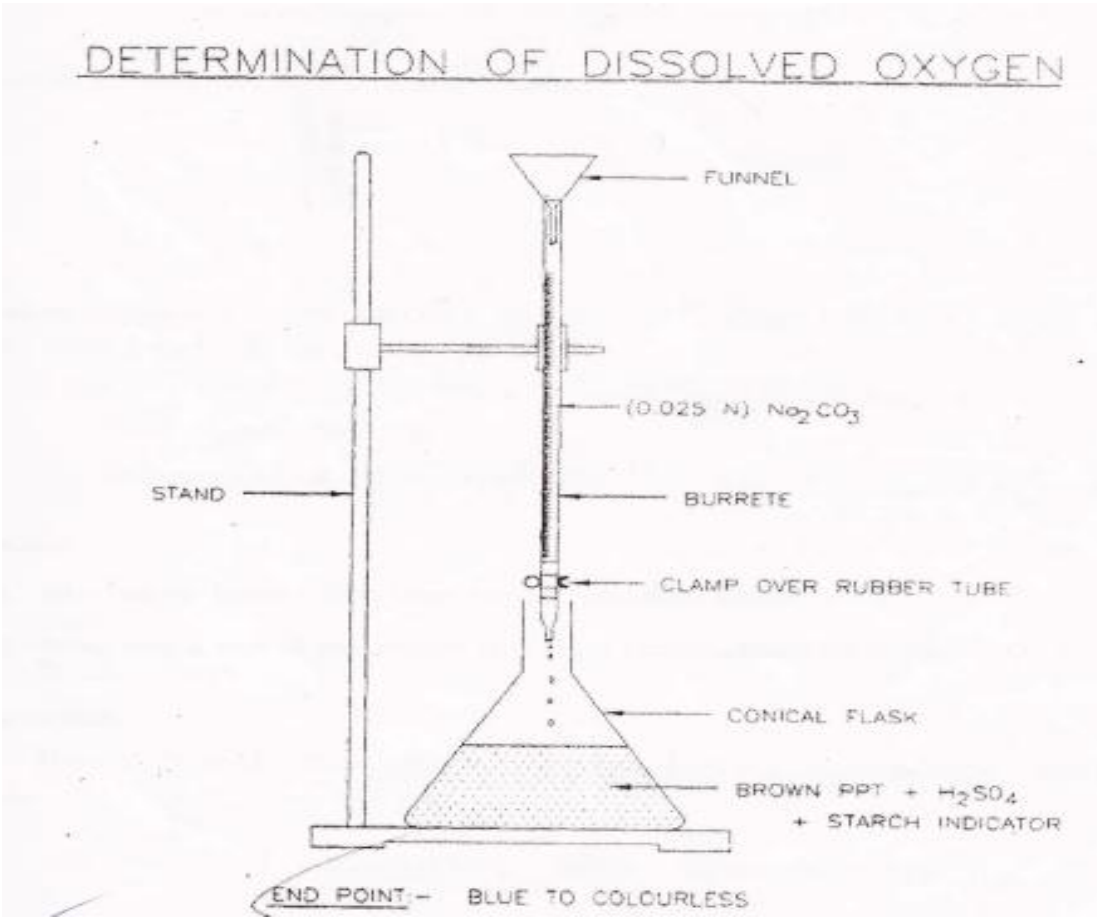
Procedure:

1. Take sample of water in BOD bottle of 300 ml.
2. Add 2 ml of manganese sulphate solution & 2 ml of potassium iodide solution.
3. Add 2 ml concentrated sulphuric acid to dissolve the precipitate.
4. Then take 200 ml of this mix in a conical flask.
5. Add starch indicator in it, colour becomes blue to colourless.

Observation Table:

Sr. No	Description	Burette reading, ml			Dissolved Oxygen, Mg/l
		I.R	F.R.	Diff.	
1.					
2.					

Diagram:



Calculation:

When whole contents have been titrated Dissolved Oxygen (mg/l) is given by,

$$\text{Dissolved oxygen} = \frac{(\text{ml x N}) of\ titrate\ x\ B}{V1 - V} \quad \text{x 1000 mg/l}$$

When only a part of the contents have been titrated,

$$\text{Dissolved oxygen} = \frac{(\text{ml x N}) of\ titrate\ x\ B}{\text{V1 x } \frac{(V1^2 - V)}{V!}} \quad \text{X 1000 mg/l}$$

For whole contents have been titrated & KI added,

Dissolved Oxygen =

Results:

1. When whole content have been titrated, Dissolved Oxygen (DO) = ...…… mg/l
2. When only a part of the content have been titrated, Dissolved Oxygen (DO) = ……..mg/l

Conclusion:

Calculation:

Experiment No: 12 (A)

Determination of BOD

Aim: - To determine Biochemical oxygen demand of given waste water sample.

Apparatus:-

Incubator, air compressor, 300ml BOD bottles with stoppers, Burette, pipette, measuring cylinders, conical flask.

Reagent:-

Sodium thiosulphate, Potassium iodide azide, manganese sulphate, conc. Sulphuric acid, starch indicator, ferric chloride, phosphate buffer, magnesium sulphate ammonium chloride etc.

Theory:-

Biochemical oxygen demand is defined as the amount of oxygen required by bacteria to decompose the organic matter under aerobic conditions. The test is one of the most important in stream pollution control activities. This is essentially a bioassay procedure involving measurement of oxygen consumed to stabilize the waste under condition as similar to those occur in nature.

Because of limited solubility of oxygen, strong, waters must be diluted to a level to ensure dissolved oxygen is present throughout the test to ensure uniform population of microorganism in various dilution, the dilution water is seeded with domestic wastewater. The test is performed at 20^0c which is fairly medium temperature of natural streams.

Reasonably large percentage of BOD 70-80 % is exerted during 5 days. Also to minimize the interference from oxygen oxidation of ammonia the test period is restricted to 5 days.

Procedure:-

1) Take sufficient volume of distilled water in vessel.
2) Aerate the water in vessel with the help of air compressor for about one hour.
3) Add 1 ml phosphate buffer, ferric chloride, magnesium sulphate each per liter of dilution water.
4) To seed the dilution water 2 ml domestic sewage per liter of dilution water.
5) After preparation of dilution water, determination the initial D.O of blank (B1) on present day.

6) Intubate the sample with at least 2-3 dilutions & blank (at least 2 nos in the incubator at 20^{0} for 5 days.
7) At the end of days determine the DO of blanks & note down their average value of as DO of blanks on 5^{th} days.
8) Determine the DO of sample as D2 for each dilution after 5 days.
9) By taking different in 0^{th} days valves & 5^{th} day's valves with dilution factor determine the BOD of sample for each dilution.
10) take the average of BOD valves of different dilution as BOD of given sample.

Environmental Significance:-

BOD test is widely used to determine the pollution AL strength of domestic as well as industrial wastes.

This test is of prime important to evaluate the purification capacity of water bodies.

BOD is an important parameter used in design of biological treatment facilities. It is also used to access the efficiency of treatment facilities.

Observation:-

i) Initial DO of blank B1 =------
ii) Final DO of blank B2 =---------

iii) Initial DO of sample (1st dilution) D1= -----
iv) Final DO of sample (1st dilution) D2=------

v) Initial DO of sample (2nd dilution) D1= -----
vi) Final DO of sample (2^{nd} dilution) D2=------

Calculation:-

Dissolved oxygen (mg/lit) = Burette reading x n x 1000/ volume of sample

Result:-

The biochemical oxygen demand of given wastewater sample is ……. mg/lit.

Experiment No. 12 (B)

Determination of COD

Introduction: - COD is a Measure of the total quantity of oxygen required for oxidation of nearly all organic compounds in wastewaters, by the action oil a strong oxidizing agent. During the test, $k_2cr_2o_7$ is used as the chemical oxidizing agent, as it can oxidize a very large variety of organic substances into Co_2 & H_2o.Aromatic hydrocarbons and pyridine are exceptions- they remain unoxidized.

BOD is a measure of only the carboneous component of biodegradable organic matter a in waste, whereas COD measures nearly all the oxidizable matter in a waste. Therefore COD for a waste is greater than its BOD value. Both BOD & COD values of any waste are important parameters, as their inter-relationship decides the type of treatment to be adopted for the waste. If COD is very much greater than BOD, then the waste is not biodegradable. Biodegradability of the waste is indicated by the treatability index.

Chemically oxidizable matter

(Estimated by COD test)

- Inorganic oxidizable matter

  (Cyanide, ferrous compounds, Nitrites, Chlorides, sulphides etc.)
- Organic matter
  - Non-biodegradable (Cellulose, lignin)
  - Biodegradable

    Nitrogenous

    Carbonaceous (Estimated by BOD test)

Importance of COD Test:-

l) COD value indicates practically overall pollution strength of a raw waste, domestic or indusial.

2) COD and BOD values of a waste are used for determining its treatability index.

3) COD test is used for quickly evaluating performance-efficiency of treatment units and correcting error immediately, as the test can be completed in 3 hours as against 5 days or 3 required for BOD test.

4) By knowing the general COD/BOD ratio for a waste, BOD values can be worked out in an emergency from COD test results.

5) COD test is used for determining the suitability of a treated waste for disposal.

Standards Recommended	
Effluents	Max. permissible COD
1) Industrial effluents discharged into inland surface water.	250 mg/l, BIS 2490 250 mg/l, CPCB
2) Industrial effluents falling into coastal areas	250 mg/l, BIS 7968 250 mg/l, CPCB

Limitations of' COD Test:

The test adopts an artificial procedure. It cannot differentiate between biologically degradable and biologically resistant organic matter, whereas BOD test simulates condition obtainable in a natural Stream.

COD test does not indicate time - wise, the rate and extent of removal of pollution load of water in nature; whereas BOD_1, BOD_2, BOD_3 to BOD_{20} indicate the natural rate of biodegradation.

Determination of COD

Principle: - A known volume and normality of a potent oxidizing agent is used to oxidize all the oxidizable matter in the waste sample as. Completely as possible. Oxidation is carried out for an extended period at l00^{o}c. The residual oxidizing agent estimated using a suitable reducing agent. The amount of oxidizing agent consumed is a measure of the overall pollution load of the waste.

Discussion:

Oxidizing agent:-

The oxidizing chemical used is $k_2cr_2o_7$ it is readily available in pure analytical grad and the standards prepared retain their normality for very long. Acidified dichromate oxidize nearly all types of organic matter into CO2 of water.

$$C_6H_{12}0_6 + (4K_2Cr_2O_7 + \text{excess of } K_2Cr_2O_7) + 16H_2SO_4 \rightarrow$$
$$6\ CO_2 + 22H_2O + 4Cr2\ (SO_4)_3 + 4k_2(SO_4) + (\text{excess of } K_2Cr_2O_7)$$

The colour of dichromate being orange helps in recognizing the presence of residual dichromate after heating, which is essential for the success of the tut. Dichromate consumed during the test is the difference between the total dichromate concentration at the start and the dichromate conc. remaining as excess after hot digestion. The initial conc. Of dichromate is estimated by running a blank through the test, which will practically eliminate error due to any oxidizable matter present in dilution water.

Reducing Agent:-

The reducing agent which is used as a titrant, is ferrous ammonium sulphate. The Ferrous ion reduces dichromate completely and excess of Fe^{++} gives a sharp reddish brown end point with Ferroin indicator.

$6\ Fe\ (NH_4)\ (SO_4)_2 + K_2Cr_2O_7 + 7H_2SO_4 \dashrightarrow 3\ Fe_2\ (SO_4)_3 + 7H_2O + 6(NH_4)_2SO_4 +$

COD of a Waste water sample, mg/lit=

$$\frac{(a-b) \times \text{Normality of titrant} \times \text{equivalent wt. of oxygen} \times 1000 \times \text{dilution factor}}{\text{Volume of sample}}$$

taken a = ml. of titrant used for blank

b = ml. of titrant used for sample (directed or diluted)

Volume of sample taken is generally 20 ml. (directed or diluted)

It is conventional to use 0.25 N $k_2Cr_2O_7$, as each ml of dichromate is equivalent to 2 mg of oxygen and so a large range of COD is determinable.

The titrant $Fe(NH_4)(SO_4)_2$ is prepared to be 0.lN so that, in an ideal case, 25ml will be required to reduce 10ml of 0.25N $K_2Cr_2O_7$ in the blank, when sufficient reaction time is allowed, for the complete reduction of dichromate (Cr^{6+}) to chromate (Cr^{3+}).

Importance of HgS04

$HgS0_4$ is used during the test to prevent the interference due to chlorides in wastewater. Chlorides reduce dichromate (Cr^{6+}) in an acidic medium, thus resulting in a higher COD value.

$$6NaCl + K_2Cr_2O_7 + 7H_2SO_4 \rightarrow 3Cl2 + 7H_2O + 3Na_2SO_4 + Cr_2\ (SO_4)_3 + K_2SO_4$$

If $HgSO_4$ is present, it combines with chlorides to form poorly ionized $HgCl_2$ thus preventing

$$HgSO_4 + 2NaCl \rightarrow HgCl_2 + NaSO_4$$

$HgSO_4$ should be added to the sample before addition of dichromate and acid 400mg of $HgSO_4$ added 20ml, of sample interference due to 4780 mg/lit of chloride concentration in sample, which is generally not executed in inland wastewater.

However, if seawater infiltration is suspected use of 1 to 2 g of $HgSO_4$ may be necessary to suppress interference due to chloride in the range of 11,000 mg/lit to 23,000 mg/lit in sample.

Importance of $AgSO_4$

$AgSO_4$ is a catalyst which enable dichromate to oxidize low molecular fully acid & straight chain allopathic compounds.

Ferroin (Ferrous 1, 10- phenantholine sulphaie)

This is a soluble organic indicator, which exists in two difference colours. The change in colour occurs with a change in oxidation- reduction potential.

In the first state, when the oxidizing agent is in excess, the colour of the indicator merges with the colour of dichromate. As titration progresses, orange dichromate (Cr^{6+}) is reduced and green chromate (Cr^{3+}) increases. At the end point, when all the dichromate ions are completely reduced addition of a very little excess of titrant makes ferrous ions available to 1, 10 – phenantholine in ferroin to form reddish – brown complex.(the colour and composition of the indicator itself

Apparatus and equipnrent:-

50ml. burratte, 10ml. pipette, 100 ml measuring cylinder, 250ml beaker, and reflux apparatus (coiled condensers attached to 250ml COD flasks with ground glass necks, mounted on heating equipment.)

Chemicals:-

1) 0.25N potassium dichromate ($K_2Cr_2O_7$ (12.259) g/lit.) - oxidizing agent.
2) conc. H_2So_4 (36N) - provides low PH necessary for oxidation by dichromate.
3) 0.lN Ferrous ammonium sulphate (Fe $(NH_4)_2$ $(SO_4)_2$ $6H_2O$ (39 g) + $36NH_2SO_4$ (20ml.) /lit. 4) Ferroin [1, 10- phenonthroline monohydrate (1.735 g; 1 $feSO_4$ $7H_2O$ (0.695G) / 100rn1 acidified.
5) Mercuric sulphate (analytical crystals)
6) Silver sulphale (reagent powder)

Standardization of Ferrous Ammonium Sulphate Necessity:-

Ferrous Ammonium Sulphate, being a reducing agent, is slowly oxidized by the oxygen dissolved from the air - from the moment the titrant is prepared. The strength, of the titrant gradually drops on standing and exposure. Therefore Standardization is required every time the titrant is used.

Procedure:-

A) 1) Pipette out 10 ml. of 0.25N K2Cr2O7 accurately into a volumetric flask and dilute to l00ml. (Using distilied water). Transfer to a 25ml conical flask.

2) Add 30rnl. of conc. H_2SO_4 (30N)

3) – Cool to room temp. (This is verf important.

4) Add 2 or 3 drops of Ferroin indicator.

5) Titrant against the given Ferrous Ammonium sulphate.

6) Record the amount of titrant (X ml.) used up to the end point, when the contents change from dark green to stable-brown colour.

Fe $(NH_4)_2$ $(SO)_2$ =

$K_2Cr_2O_7$ N x EX (m1) = 0.025 x

100 (ml.) 0.025 x 100 ml.

Normality of titrant (N) $\frac{0.025 \text{ x } 100\text{ml}}{\text{X ml}}$ =

Procedure of COD test:-

A) 1) Take three COD flasks P, Q, and R and place about 400 mg (by empirical estimate of $HgSO_4$) in each flask.

2) Add 20ml.of distilled water to flask P (blank flask) and 20ml of sample direct or Diluted to flasks Q and R (Adopt two different dilutions. For flasks Q and R) 3) Add 10ml of .25N $K_2Cr_2O_7$ accurately, using a pipette, to each flask.

4) Add 30m1 of conc- H_2SO_4 (36 N) to each flask slowly in installments, stirring the contents carefully.

5) Add about 200mg of Ag_2SO_4 to each flask.

6) Add 3 to 4 glass beads or rounded quartz pebbles (to bumping of acid mixture during boiling)

B) 1) Attach all the free flasks to Reflux condensers. Heat and digest for two hours.

2) Cool the flasks. Add 20ml of distilled water down each condenser attached to P, Q, & R (to wash down concensed organics sticking to coiled surface) 3) Detach the flasks and add 70mldistilled water to each.

4) cool the flasks to room temperature- (This is very important. If the flask contents are at higher temp. Than the titrant, then very large quantities of titrant will be used and COD test will be a failure.)

C) l) Titrate all the three flasks against standardized Ferrous ammonium sulphate using 2 To 3 drops of Ferroin indicator.

2) Record titrant used - a) ml. for blank flask. P & (b) ml for sample flask Q and (c) ml. for sample flask R.

Result : The amount of COD determined from the provided water sample is _____ mg/l

Preparation of Reagents and Media

Reagents for various determinations are prepared as follows:

Alkalinity

1. **0.02 N standard sulphuric acid:** Prepare stock solution approximately 0.1 N by diluting 2.5 mL concentrated sulphuric acid to 1 litre. Dilute 200 mL of the 0.1 N stock solution to 1 litre CO_2 free distilled water. Standardise the 0.02 N acid against a 0.02 N sodium carbonate solution which has been prepared by dissolving 1.06 g anhydrous Na_2CO_3 and diluting to the mark of a 1 litre volumetric flask.
2. **Methyl orange indicator:** Dissolve 500 mg methyl orange powder in distilled water and dilute it to 1 litre. Keep the solution in dark or in an amber coloured bottle.
3. **Phenolphthalein indicator:** Dissolve 5 g phenolphthalein in 500mL ethyl alcohol and add 500 mL distilled water. Then add 0.02 N sodium hydroxide drop-wise until a faint-pink colour appears.
4. **Sodium thiosulphate 0.1 N:** Dissolve 25 g $Na_2S_2O_3.5H_2O$ and dilute to 1 litre.

Hardness

5. **Ammonia buffer solution:** Dissolve 16.9 g ammonium chloride (NH_4Cl) in 143 mL concentrated ammonium hydroxide (NH_4OH). Add 1.25 g magnesium salt of EDTA and dilute to 250 mL with distilled water. Do not store more than a month's supply. Discard the buffer when 1 or 2 mL added to the sample fails to produce a pH of 10.0±0.1 at the end point of titration. Keep the solution in a plastic or resistant glass container.
6. **Eriochrome black T indicator:** Mix 0.5 g Eriochrome black T dye with 4.5 g hydroxylamine hydrochloride. Dissolve this mixture in 100 ml of 95% ethyl or isopropyl alcohol.
7. **Standard EDTA titrant 0.01 M:** Weigh 3.723 g analytical reagent grade EDTA disodium salt ($Na_2H_2C_{10}H_{12}O_8N_2$) and dissolve in distilled water and dilute to 1litre.

Chloride

8. **Potassium chromate indicator:** Dissolve 50 g potassium chromate ($K_2Cr_2O_4$) in a little distilled water.Add silver nitrate solution until a definite red precipitate is formed. Let stand for 12 hours, filter and dilute the filtrate to 1 litre with distilled water.
9. **Standard silver nitrate solution 0.0141 N:** Dissolve 2.395 g $AgNO_3$ in distilled water and dilute to 1 litre. Standardise against 0.0141 N NaCl. Store in a brown bottle; 1 mL = 500 µg C_{l2}.
10. **Standard sodium chloride 0.0141N:** Dissolve 824.1 mg NaCl (dried at 140°C) in chloride free water and dilute to 1 litre. 1mL = 500 µg C_{l2}.
11. **Aluminium hydroxide suspension:** Dissolve 125 g aluminium potassium sulphate in 1 litre water. Warm to 60°C and add 55 mL concentrated NH4OH slowly with stirring. Let stand for

1 hour, transfer the mixture to a large bottle. When freshly prepared the suspension occupies a volume of approximately 1 litre.

Iron

12. **Hydrochloric acid:** Concentrated HCl.
13. **Hydroxylamine solution:** Dissolve 10 g hydroxylamine hydrochloride salt ($NH_2OH.HCl$) in 100 mL distilled water.
14. **Ammonium acetate buffer solution:** Dissolve 250 g ammonium acetate ($NH_4C_2H_3O_2$) in 150 mL distilled water. Add 700 mL concentrated (glacial) acetic acid.
15. **Sodium acetate solution:** Dissolve 200 g sodium acetate ($NaC_2H_3O_2.{}_3H_2O$) in 800 mL distilled water.
16. **Phenanthroline solution:** Dissolve 100 mg 1, 10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$) in 100 mL distilled water by stirring and heating to 80°C. Do not boil. Discard the solution if it darkens. Heating is unnecessary if 2 drops of concentrated HCl are added to the distilled water. 1 mL of this reagent is sufficient for no more than 100 µg Fe.
17. **Stock iron solution:** Add slowly 20 mL concentrated H2SO2 to 50 mL distilled water and dissolve 1.404 g ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2.6H_2O$]. Add 0.1 N KMnO4 drop wise until a faint-pink colour persists. Dilute to 1litre with iron free distilled water. Each 1 mL of this solution contains 200 µg Fe.
18. **Standard iron solution:** Pipette 50 mL stock solution into 1 litre volumetric flask and dilute to the mark with distilled water. 1 mL = 10 µg Fe.

Manganese

19. **Special reagent:** Dissolve 75 g mercuric sulphate ($HgSO_4$) in 400 mL concentrated nitric acid (HNO_3) and 200 mL distilled water. Add 200 mL 85% phosphoric acid and 35 mg silver nitrate to the above solution. Dilute the cooled solution to 1 litre.
20. **Ammonium persulphate:** $(NH_4)_2S_2O_3$ solid.
21. **Standard manganese solution:** Prepare a 0.1 N potassium permanganate ($KMnO_4$) solution by dissolving 3.2 g of $KMnO_4$ in distilled water and making it up to 1 litre. Age for several days in sunlight or heat for several hours near the boiling point and then filter through fritted glass filter crucible and standardise against sodium oxalate. Calculate the volume of this solution necessary to prepare 1 litre solution of such strength that 1mL = 50 µg Mn as follows:

$$KMnO_4 = 4.55 \div \text{Normality of } KMnO_4 mL$$

To this solution add 2 to 3 mL concentrated H_2SO_4 and sodium bisulphite solution (10 g $NaHSO_3$ + 100 mL distilled water). Boil to remove excess SO_2, cool and dilute to 1000 mL with distilled water.

Sulphate

22. **Conditioning reagent:** Mix 50 mL glycerol with a solution containing 30 mL concentrated HCl, 300 mL distilled water, 100 mL 95% ethyl or isopropyl alcohol and 75 g NaCl.
23. **Barium chloride:** Barium chloride crystals.
24. **Standard sulphate solution:** Prepare a standard sulphate solution such that 1 mL = 100 µg SO_4. Dissolve 147.9 mg anhydrous Na_2SO_4 in 500 mL distilled water and dilute to 1 litre 1 mL = 100 µg SO_4.

Sulphide

25. **Hydrochloric acid:** Prepare a 6 N solution.
26. **Standard iodine solution 0.025 N:** Dissolve 20 - 25 g potassium iodide in a little water and add 3.2 g iodine. After the iodine has dissolved, dilute to 1 litre and standardise against 0.025 N sodium thiosulphate using starch as indicator.
27. **Standard sodium thiosulphate 0.025 N:** Dissolve 6.205 g sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in freshly boiled and cooled distilled water and dilute to 1litre. Preserve by adding 5mL chloroform or 0.4 g NaOH/L or 4 g borax and 5 - 10 mg HgI_2/L. Standardise this with 0.025 N potassium dichromate solution which is prepared by dissolving 1.226 g potassium dichromate in distilled water and diluted to 1 litre.
28. **Starch indicator:** Add cold water suspension of 5 g soluble starch to approximately 800 mL boiling water with stirring. Dilute to 1 litre, allow to boil for a few minutes and let settle overnight. Use supernatant liquor. Preserve with 1.25 g salicylic acid/litre or by the addition of a few drops of toluene.

Dissolved oxygen

29. **Manganous sulphate solution:** Dissolve 480 g $MnSO_4.4H_2O$, 400 g $MnSO_2.2H_2O$ or 364 g $MnSO_4.H_2O$ in distilled water, filter and dilute to 1 litre.
30. **Alkali-iodide-azide reagent:** Dissolve 500 g NaOH or 700 g KOH and 135 g NaI or 150 g KI in distilled water and dilute to 1 litre. Add 10 g sodium azide (NaN_3) dissolved in 40 mL distilled water. The reagent should not give colour with starch when diluted and acidified.
31. **Sulphuric acid concentrated:** 1mL is equivalent to about 3 mL alkali-iodide-azide reagent.
32. **Standard sodium thiosulphate 0.025 N:** Dissolve 6.205 g sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in freshly boiled and cooled distilled water and dilute to 1 litre. Preserve by adding 5 mL chloroform or 0.4 g NaOH/L or 4 g borax and 5 - 10 mg HgI2/L. Standardise this with 0.025 N potassium dichromate solution which is prepared by dissolving 1.226 g potassium dichromate in distilled water and diluted to 1 litre.
33. **Standard potassium dichromate solution 0.025 N:** A solution of potassium dichromate equivalent to 0.025 N sodium thiosulphate contains 1.226 g/L $K_2Cr_2O_7$. Dry $K_2Cr_2O_7$ at 103°C for 2 hrs before making the solution.
34. **Standardisation of 0.025 N sodium thiosulphate solution:** Dissolve approximately 2 g KI in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 10 mL of H_2SO_4, followed by exactly 20 mL, 0.1 N potassium dichromate solution. Place in the dark for 5 minutes, dilute to approximately 400 mL and titrate with 0.025 N sodium thiosulphate solution, adding starch towards the end of titration. Exactly 20 ml 0.025 N thiosulphate will be consumed at the end of the titration. Otherwise, the thiosulphate solution should be suitably corrected.

35. **Starch Indicator:** Add cold water suspension of 5 g soluble starch to approximately 800 mL boiling water with stirring. Dilute to 1 litre, allow to boil for a few minutes and let settle overnight. Use supernatant liquor. Preserve with 1.25 g salicylic acid/1 litre or by the addition of a few drops of toluene.

BOD

36. **Phosphate buffer solution:** Dissolve 8.5 g potassium dihydrogen phosphate (KH_2PO_4), 21.75 g dipotassium hydrogen phosphate (K_2HPO_4), 33.4 g disodium hydrogen phosphate heptahydrate ($Na_2HPO_4.7H_2O$) and 1.7 g NH_4Cl in about 500 ml distilled water and dilute to 1 litre. The pH of this buffer should be 7.2 without further adjustment. Discard the reagent if there is any sign of biological growth in the stock bottle.
37. **Magnesium sulphate solution:** Dissolve 22.5 g $MgSO_4.7H_2O$ in distilled water and dilute to 1 litre.
38. **Calcium chloride solution:** Dissolve 27.5 g anhydrous CaC_{l2} in distilled water and dilute to 1 litre.
39. **Ferric chloride solution:** Dissolve 0.25 g $FeC_{l3}.6H_2O$ in distilled water and dilute to 1 litre.
40. **Sodium sulphate solution 0.025 N:** Dissolve 1.575 g anhydrous Na_2SO_3 in 1 litre distilled water. This is to be prepared daily.
41. **Seeding:** The standard seed material is settled domestic wastewater that has been stored at 20°C for 24 to 36 hours. A seed concentration of 1-2 mL/L is usually adopted.

Residual chlorine (OTA)

42. **Dissolve 1.35 g orthotolidine dihydrochloride in 500 mL distilled water:** Add this solution with constant stirring to a mixture of 350 mL distilled water and 150 mL concentrated hydrochloric acid. Store the solution in brown bottle. Always use an automatic, dropping or safety pipette to measure the necessary volume. Avoid inhalation or exposure to the skin.

Coliform test

43. **Lactose broth:** Beef extract 3 g, peptone 5 g, lactose 5 g and reagent grade distilled water 1 litre. Add these ingredients to reagent grade distilled water, mix thoroughly and heat to dissolve. pH should be 6.8 - 7.0 after sterilisation.
44. **Lauryl tryptose broth:** Tryptose 20 g, lactose 5 g, K_2HPO_4 2.75 g, KH_2PO_4 2.75 g, NaCl 5 g, sodium lauryl sulphate 0.1 g, reagent grade distilled water 1 litre, sterilise and use. Add dehydrated ingredients to water, mix thoroughly and heat to dissolve. pH should be 6.8 ±2 after sterilisation.
45. **Endo agar:** Peptone 10 g, lactose 10 g, K_2HPO_4 3.5 g, agar 15 g, sodium sulphite 2.5 g, basic fuchsin 0.5 g, distilled water 1 litre, pH 7.4 after sterilisation.
46. **EMB agar:** Peptone 10 g, lactose 10 g, K_2HPO_4 2 g, agar 15 g, eosin 0.4 g, methylene blue 0.065 g, distilled water 1 litre, pH should be 7.1 after sterilisation.
47. **Brilliant green lactose bile broth:** Peptone 10 g, lactose 10 g, oxgall 20 g, brilliant green 0.0133 g, distilled water 1 litre, pH should be 7.2 after sterilisation and is then ready for use. Store away from direct sunlight to extend the reagent stability to 6 months.

Acidity

48. **NaOH solution 0.02 N:** Dissolve 4 g NaOH in 1 litre water. This gives 0.1 N NaOH solution. Take 200 ml of this 0.1 N solution and make it up to 1 litre to obtain 0.02 N NaOH solution.
49. **Methyl orange indicator:** Dissolve 500 mg methyl orange powder in distilled water and dilute it to 1 litre.
50. **Phenolphthalein indicator:** Dissolve 5 g phenolphthalein disodium salt in distilled water and dilute to 1 litre.
51. **Sodium thiosulphate 0.1 N:** Dissolve 25 g $Na_2S_2O_3.5H_2O$ and dilute to 1 litre distilled water.

COD

52. **Standard potassium dichromate solution 0.25 N:** Dissolve 12.259 g $K_2Cr_2O_7$primary standard grade previously dried at 103°C for 2 hours and dilute to 1 litre.
53. **Sulphuric acid reagent:** Concentrated H_2SO_4 containing 22 g silver sulphate per 4 kg bottle.Dissolve 22 g Ag_2SO_2 in 4 kg bottle and keep it for 2 days. This is the reagent.
54. **Standard ferrous ammonium sulphate 0.1 N:** Dissolve 39 g $Fe(NH_4)_2(SO4)_2.6H_2O$ in distilled water. Add 20 mL conc. H_2SO_4 and cool and dilute to 1 litre. Standardise this against the standard dichromate solution. Dilute 10 mL standard $K_2Cr_2O_7$ solution to about 100 mL. Add 30 mL conc. H_2SO_4 and cool. Titrate with ferrous ammonium sulphate titrant using 2 - 3 drops of ferroin indicator.

Ammonia N

55. **Zinc sulphate solution:** Dissolve 100 g $ZnSO_4.7H_2O$ and dilute to 1 litre.
56. **EDTA reagent (stabiliser):** Dissolve 50 g EDTA disodium salt in 60 mL of water containing 10 g NaOH.
57. **Nessler's reagent:** Dissolve 100 g HgI_2 and 70 g KI in a small quantity of water and add this mixture slowly with stirring to a cool solution of 160 g NaOH in 500 mL water. Dilute to 1 litre and store in rubber stoppered pyrex glass out of sunlight.
58. **Stock ammonia solution:** Dissolve 3.811 g anhydrous NH4Cl dried at 100°C in water and dilute to 1 litre. 1 mL = 1.00 mg N and 1.22 mg NH_3.

Nitrate N

59. **Stock nitrate solution:** Dissolve 721.8 mg anhydrous potassium nitrate and dilute to 1 litre with distilled water. 1 mL = 0.1 mg N.
60. **Standard nitrate solution:** Dilute 10 mL stock nitrate solution to 1 litre. 1 mL = 1 µg N
61. **Sodium arsenite solution:** Dissolve 5.0 g $NaAsO_2$ and dilute to 1 litre.
62. **Brucine-sulphanilic acid solution:** Dissolve 1 g brucine sulphate and 0.1 g sulphanilic acid in about 70 mL of hot distilled water. Add 3 mL conc. HCl, cool and make up to 100 mL. This is stable for several months.

63. **Sulphuric acid solution:** Carefully add 500 mL conc. H_2SO_4 to 125 mL distilled water and cool to room temperature.
64. **Sodium chloride solution:** Dissolve 300 g NaCl and dilute to 1litre with distilled water.

Nitrite N

65. **Sulphanilamide reagent:** Dissolve 5 g sulphanilamide in a mixture of 50 mL conc. HCl and about 300 mL distilled water. Dilute to 500 mL with distilled water.
66. **N-(1-naphthyl)-ethylenediamine dihydrochloride solution:** Dissolve 500 mg dihydrochloride in 500 mL distilled water. Store in a dark bottle.
67. **Hydrochloric acid: HCl (1+3)**
68. **Stock nitrite solution:** Dissolve 1.232 g $NaNO_2$ in nitrite free water and dilute to 1 litre. Fresh nitrite from bottle should be taken 1 mL = 250 mg N in the solution. Preserve with 1 mL chloroform.
69. **Standard nitrite solution:** Standardise stock solution. Pipette 50 ml standard 0.05 N $KMnO_4$, 5 mL conc.H_2SO_4 and 50 mL stock nitrite solution in a glass stoppered flask. Discharge the permanganate colour by ferrous ammonium sulphate solution of 0.05 N (19.607 g ferrous ammonium sulphate and 20 mL conc.H_2SO_4 in 1 litre) strength. Carry nitrite free blank through the entire procedure and make necessary corrections. Calculate the nitrite N content of stock solution by the following equation:

$$A = [(B \times C) - (D \times E)] \times 7/F$$

where,

1. A = mg/mL nitrite N in stock solution,
2. B = total mL standard $KMnO_4$ used,
3. C = normality of $KMnO_4$ solution,
4. D = total mL of standard $Fe(NH_4)_2(SO_4)_2$ used,
5. E = normality of standard $Fe(NH_4)_2(SO_4)_2$,
6. F = mL of stock $NaNO_2$ solution taken for titration.

Each 1 mL of 0.05 N $KMnO_4$ consumed by the nitrite corresponds to 1.729 µg $NaNO_2$ or 350 µg N.

Organic Nitrogen (to find Kjeldahl Nitrogen)

70. **Digestion reagent:** Dissolve 134 g K_2SO_4 in 650 mL ammonia free distilled water and 200 mL conc.H_2SO_4. Add with stirring a solution prepared by dissolving 2 g red mercuric oxide (HgO) in 25 mL 6N H_2SO_4. Dilute the combined solution to 1 litre.
71. **Sodium hydroxide-sodium thiosulphate reagent:** Dissolve 500 g NaOH and 2 g $Na_2S_2O_3.5H_2O$ in ammonia free distilled water and dilute to 1 litre.
72. **Borate buffer solution:** Add 88 mL 0.1N NaOH solution to 500 mL 0.025 M sodium tetraborate ($Na_2B_4O_7$) solution (5 g $Na_2B_4O_7$ in 1 litre) and dilute to 1 litre.
73. **Sodium hydroxide 6 N:** Dissolve 240 g NaOH in 1 litre ammonia free distilled water.
74. **Standard iodine 0.1 N:** Dissolve 40 g KI in 25 ml distilled water, add 13 g resublimed iodine and stir until dissolved. Transfer to 1 litre volumetric flask and dilute to the mark.

Fluoride

75. **Standard fluoride solution:** Dissolve 221 mg anhydrous sodium fluoride in distilled water and dilute to 1 litre. 1 mL = 100 µg F. This is the stock solution. Pipette 100 mL stock solution and make it up to 1 litre with distilled water to obtain standard solution 1ml = 10 µg F.
76. **Zirconyl-alizarin reagent:** Dissolve 300 mg zirconyl chloride octahydrate ($ZrOC_{l2}.8H_2O$) in 50 mL distilled water contained in 1 litre glass stoppered volumetric flask. Dissolve 70 mg of 3-alizarin sulphonic acid sodium salt (also called alizarin red S) in 50 mL distilled water and pour slowly into the zirconyl solution while stirring. The resulting solution clears on standing for a few minutes.
77. **Mixed acid solution:** Dilute 101 mL conc. HCl to approximately 400 mL with distilled water. Add carefully 33.3 mL conc. H_2SO_4 to approximately 400 mL distilled water. After cooling, mix the two acids.
78. **Acid-zirconyl-alizarin reagent:** To the clear zirconyl-alizarin reagent in 1 litre volumetric flask, add the mixed acid solution and distilled water to the mark and mix. The reagent changes in colour from red to yellow within an hour.