

Experiment No.1

Determination of Solids in Sewage

AIM:

To determine the total solids, Dissolved Solids and Suspended solids in the given sample.

THEORY:

The term solids refers to the matter either filterable, in filterable that remains as residue upon evaporation and subsequent drying at a defined temperature. Further categorization depends upon the temperature employed for drying and ignition. Different forms of solids are defined on the basis of method applied for their determination. All solids are measured gravimetrically except settle-able solids by volume and total dissolved solids by specific conductance.

APPARATUS:

- 1) Electronic Balance
- 2) Crucibles
- 3) Filter Paper
- 4) Heater

PROCEDURE:

Total Solids:

1. The crucibles are cleaned with water and the empty weights of the crucibles are taken (W_1).
2. About 50 ml of sample is taken and transfer it into the crucible.
3. The crucible is then placed in an electrical oven at 103^0 to 105^0 C. and sample is made to evaporate till the residue is obtained.
4. The weight of crucible with residue is taken.(W_2)

$$\text{Total Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

Dissolved Solids:

1. The crucibles are cleaned with water and the empty weights of the crucibles are taken (W_1).
2. About 50 ml of sample is taken and filtered through a filter paper. The sample is then transferred into crucible.

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3. The crucible is then placed in an electrical oven at 103⁰ to 105⁰ C. and sample is made to evaporate till the residue is obtained.
4. The weight of crucible with residue is taken.(W₂)

$$\text{Dissolved Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

Suspended Solids:

Suspended solids are those which are in suspension in solution. The suspended solids are removed by filtration. The residue left on the filter paper is the suspended solid and it is determined by drying the filter paper at 103⁰ to 105⁰ C.

Suspended solids are also determined as,

$$\text{Suspended solids} = \text{Total solids} - \text{Dissolved solids.}$$

OBSERVATIONS:

For Tap water (without filter)

Quantity of sample taken =

Empty wt. of crucible = W₁ =

Wt. of crucible + Residue = W₂ =

$$\text{Total Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

For Tap water (filtered)

Quantity of sample taken =

Empty wt. of crucible = W₁ =

Wt. of crucible + Residue = W₂

$$\text{Dissolved Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

Suspended solids in Tap water = Total solids – Dissolved solids

For Sewage (without filter)

Quantity of sample taken =

Empty wt. of crucible = W₁ =

Wt. of crucible + Residue = W₂

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$$\text{Total Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

For Sewage (filtered)

Quantity of sample taken =

Empty wt. of crucible = W_1 =

Wt. of crucible + Residue = W_2

$$\text{Dissolved Solids in mg per Lit.} = \frac{(W_2 - W_1) \times 1000}{\text{ml of sample taken.}}$$

Suspended solids in Tap water = Total solids – Dissolved solids

RESULTS:-

1. Total Solids in Tap water in mg per Lit =
2. Dissolved Solids in Tap water in mg per Lit =
3. Suspended solids in Tap water =
4. Total Solids in Sewage in mg per Lit =
5. Dissolved Solids in Sewage in mg per Lit =
6. Suspended solids in Sewage =

CONCLUSION:

As per IS:10500-1991, there is no limit prescribed for total solids, but as per WHO the permissible limit for total solids is 1200mg/lit. and the limit for dissolved solids as per IS:10500 is 500 mg /lit.

Experiment No.2

Determination of Conductivity.

AIM:

To determine the conductivity of the given sample.

THEORY:

The electrical conductivity is the measure of capacity of a substance or solution to carry electrical current. The conductivity is represented by the reciprocal value of electrical resistance in ohms relative to cubic centimeter of water at 25°C. The electrical conductivity is a total parameter for dissolved and dissociated substances .Its value depends on concentrations and degree of dissociation of the ions as well as the temperature and migration velocity of the ion in the electric field.

APPARATUS AND REAGENTS:

1. Conductivity meter with measuring cell.
2. Beaker.
3. Thermometer.
4. KCl 0.1N.

PROCEDURE:

- 1 Calibrate the cell with standard 0.1N KCl solution of conductivity 14.12 mmohs at 30°C.
- 2 Rinse the cell thoroughly with deionized distilled water and carefully wipe with tissue paper.
- 3 Dip the cell into the sample solution, swirl the solution and wait up to 1 minute for a steady reading.
- 4 Note down the instrument reading and also temperature by a thermometer.

OBSERVATIONS:

| Description of sample | Temperature | Conductivity(mmohs) | Remarks |
|-----------------------|-------------|---------------------|---------|
| | | | |
| | | | |
| | | | |
| | | | |

RESULT:

The conductivity of the given sample =

CONCLUSION:

Electrical conductivity measurements are often employed to monitor desalination plants. In coastal regions conductivity data can be used to decide the extent of intrusion of sea water into the ground water .Conductivity data is useful in determining the suitability of drinking water and waste water for disposal on land .Irrigation waters up to 2 mmohs/cm conductance have been found to be suitable for irrigation depending on soils and climatic characteristics.

Experiment No.3

Determination of Sulphate content.

AIM:

To determine the concentration of sulphate in the given sample of water

THEORY:

Sulphates are found in appreciable quantity in all natural water particularly high in arid and semi-arid region. Domestic sewage and industrial effluents besides biological oxidation of reduced sulphur species may add to sulphate content of water. In the region where atmospheric sulphur content is high because of industrial and automobile emission. The rain water has high sulphur content. Sulphate salts are mostly soluble and impart hardness to water with a 500mg/lit sulphate have a bitter taste and those with 1000 mg/lit or more sulphate may cause intestinal disorders.

APPARATUS AND REAGENTS:

1. Conical flask.
2. Burette.
3. Spectrophotometer.
4. Magnetic stirrer.
5. Filter paper.
6. NaCl-HCl solution.
7. Glycerol ethanol solution.
8. Standard Sulphate solution.

PROCEDURE:

- 1) Prepare 100ml of solution by adding standard solution multiples calculated as follows from the equation

$$= \frac{X \times N \times 1000}{100}$$

(where N=Normality)

- 2) Take 100 ml of sample in a conical flask add 10 ml of NaCl-HCl solution.
- 3) To this add 10 ml glycerol ethanol solution and a pinch of barium chloride.
- 4) Keep the flask constantly stirred with the help of magnetic stirrer.

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- 5) Measure the absorbance developed on spectrophotometer at 420nm
- 6) Process the standard solution of different strengths in similar way and record the absorbance for each and plot a standard curve.
- 7) Deduce the sulphate content of the sample in mg/lit by comparing the absorbance of sample with standard curve.

OBSERVATIONS:

Wavelength used $\lambda = 420\text{nm}$

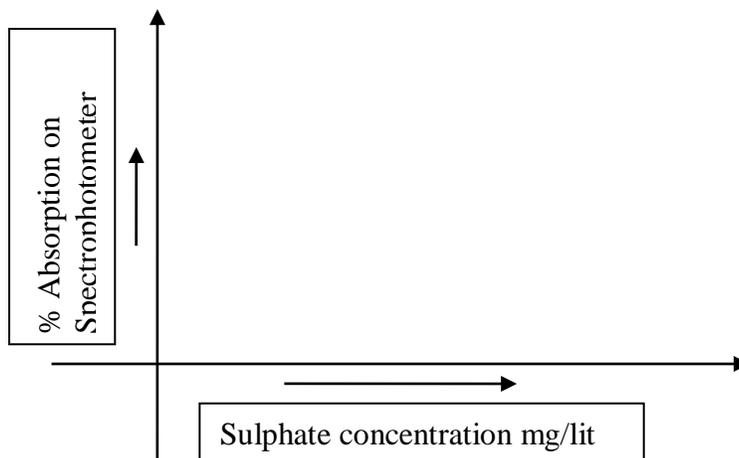
| SI No. | Concentration | Transmission |
|--------|---------------|--------------|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |

RESULT:

The concentration of sulphate in given sample of water is =

CONCLUSION:

Sulphates Na_2SO_4 and MgSO_4 should not be present in drinking water as they cause cathartic action. Higher concentration of sodium sulphate in water can cause malfunctioning of the alimentary canal. So the recommended upper limit is 250 mg/l in waters intended for human consumption.



Experiment No.4

Determination of Chloride Concentration.

AIM:

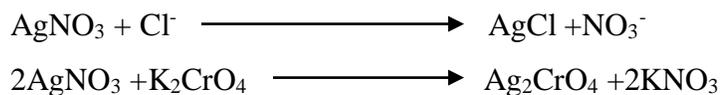
To determine the chloride ion concentration in the given sample.

THEORY:

Chloride anion is generally present in natural water. The presence of chloride in natural water can be attributed to the distribution of salts deposits discharging of effluent from chemical industries, oil well operations, Sewage discharge, irrigation discharge contamination from refuse leachates and sea water intrusion in coastal areas. Each of these sources may result in local contamination of both surface water and ground water.

The salty taste produced by chloride depends on the chemical composition of water. Concentration of 250 mg/lit may be detectable in some water containing sodium ions. Chloride in a natural or slightly alkaline solution is detected by titration with standard silver nitrate using potassium chromate as an indicator, silver chloride quantitatively precipitated before red silver chromate is formed.

Chloride ion is determined by Mohr's method, titration with standard silver nitrate solution in which silver chloride is precipitated at first. The end of titration is indicated by formation of red silver chromate from excess AgNO_3 and potassium chromate used as an indicator in neutral to slightly alkaline solution.



APPARATUS AND REAGENTS:

- 1 Burette.
- 2 Pipette.
- 3 Conical flask.
- 4 Potassium chromate solution.
- 5 Silver nitrate solution.

PROCEDURE:

- 1) Take 25 ml of distilled water in a clean conical flask add a pinch of Potassium chromate solution to the conical flask and stir the sample well.

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- 2) Titrate the solution against silver nitrate till the solution gets brick red colour.
Note down the burette reading B and it is treated as blank reading.
- 3) Take 25 to 50 ml of sample in a conical flask
- 4) Add a pinch of Potassium chromate solution to the conical flask
- 5) In case of sewage sample it must be filtered first and then the test is to be conducted.
- 6) Titrate the above sample against silver nitrate till the solution gets brick red colour that is it matches with the solution giving blank reading. Note down the reading .The colour changes from yellow to brick red

OBSERVATIONS:

| Sl No. | Sample details | Initial reading (A) | Final reading (B) | Difference | Average |
|--------|-----------------|---------------------|-------------------|------------|---------|
| 1 | Distilled water | | | | |
| 2 | Tap water | | | | |
| | | | | | |
| | | | | | |
| 3 | Sewage | | | | |
| | | | | | |
| | | | | | |

CALCULATIONS

$$\text{Chloride Content mg/l} = \frac{(A-B) \times 35.45 \times 1000 \times N}{\text{ml of Sample}}$$

RESULT:

The Chloride content in given sample of tap water is =

The Chloride content in given sample of Sewage is =

CONCLUSION:

Chlorides associated with sodium exerts salty taste when its concentration is more than 250 mg/l...Chlorides are generally limited to 250 mg/l .It can also corrode concrete. Chloride determination in natural waters is useful in the selection of water for human use. It also helps in the determination of the type of desalting apparatus to be used. It is used to control pumping of ground water from locations where intrusion of sea water is a problem.

Experiment No.5

Determination of Acidity

AIM:

To determine acidity of a given sample.

THEORY:

Acidity of a liquid is its capacity to donate H⁺ ions. Since most of the natural waters and sewages are buffered by carbon dioxide bicarbonate system, the acidity present due to free CO₂ has no significance from public health view point. Waters containing mineral acidity (due to H₂SO₄ and HCl, HNO₃) are unacceptable. Further, acid waters pose problem of corrosion and interfere in water softening. The mineral acids present and contributing mineral acidity can be calculated by titrating or neutralizing samples to pH 4.3. The CO₂ and bicarbonates (carbonic acid) present in the sample can be neutralized completely by continuing the titration to pH 8.3.

APPARATUS AND REAGENTS:

1. Burette.
2. Pipette.
3. Conical flask
4. Methyl Orange indicator
5. Phenolphthalein indicator
6. N/50 Sodium Hydroxide solution

PROCEDURE:

Place 100ml water in a conical flask and add to it one drop of methyl orange indicator. If it gives a orange red colour it means that mineral acidity is present. Hence titrate with N/50 NaOH solution to get yellow colour, that being the end point. Note the ml of N/50 NaOH solution used.

If there is no change on addition of methyl orange indicator, in another flask. Take a fresh sample and add 0.5 ml of phenolphthalein indicator. If it does not give any colour. Titrate it with N/50 NaOH to get pink colour, that being the end point. Note the ml of N/50 NaOH solution used. If phenolphthalein indicator gives a pink colour on addition in the sample. Acidity is not present.

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OBSERVATIONS:

| Sample details | Methyl orange indicator | | | Phenolphthalein indicator | | |
|--------------------------------|-------------------------|-------|---------|---------------------------|-------|---------|
| | initial | Final | reading | initial | final | Reading |
| I. Tap water 1) 2) 3) | | | | | | |
| II. Sewage 1) 2) 3) | | | | | | |

CALCULATION:

$$\text{Acidity due to mineral or CO}_2 = \frac{(A \times N \times 1000)}{\text{ml of sample}}$$

A: ml of NaOH used

N: normality of NaOH

RESULT:

Acidity due to mineral or CO₂ for the given sample =

CONCLUSION:

. Water having acidity more than 50 mg/lit cannot be used in RCC works. Water containing mineral acidity (due to H₂SO₄ and HCl, HNO₃) is unacceptable.

Experiment No.6

Determination of Alkalinity

AIM:

To determine presence of alkalinity in given sample.

THEORY:

The alkalinity of water is a measure of its capacity to neutralize acids. The alkalinity of natural water is due to salts of carbonates bicarbonates, silicates and phosphorous along with the hydroxyl ions in the free state .However the major portion of alkalinity in natural water is caused by hydroxide, carbonate, bicarbonates which may be ranked in order of their association with high pH values. Alkalinity values provide guidance in applying paper dosage of chemicals in water and waste water treatment processes particularly in coagulation softening and operational control of anaerobic digestion.

Alkalinity of sample can be estimated by treating it with standard H_2SO_4 . Titration up to pH 8.3 or decolourisation of phenolphthalein indicator will indicate complete neutralization of OH and half of CO_3 while up to pH 4.5 or sharp change from yellow to orange of methyl orange indicator will indicate total alkalinity.(Complete neutralization of OH, CO_3 , HCO_3 etc.)

APPARATUS AND REAGENTS:

1. Burette.
2. Pipette.
3. Conical flask
4. Standard H_2SO_4 of 0.02N.
5. Methyl Orange indicator.
6. Phenolphthalein indicator.

PROCEDURE:

1. Take 50ml of sample in a conical flask and add two to three drops of Phenolphthalein indicator.
2. If pink colour develops then titrate with 0.02N H_2SO_4 till it disappears or pH is 8.3.Note the volume of H_2SO_4 required (Reading A).

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3. Add two to three drops of Methyl orange to the same flask and continue titration till pH comes down to 4.5 or yellow colour changes to orange .Note the volume of H₂SO₄ added.(Reading B)
4. In case pink colour does not appear after addition of phenolphthalein continue as in step 3.
5. Colour total (T) phenolphthalein (P) Methyl orange alkalinity as follows express in mg/lit as CaCO₃.

$$P = \text{Alkalinity mg/lit as CaCO}_3 = \frac{A \times N \times 1000}{\text{ml of sample}}$$

$$T = \text{Alkalinity mg/lit as CaCO}_3 = \frac{B \times N \times 1000}{\text{ml of sample}}$$

In case H₂SO₄ is not 0.02N then apply the formula Alkalinity in mg/lit as CaCO₃

$$= (A/B) \times N \times 5000 / \text{ml of sample}$$

Where A= ml of H₂SO₄ required to bring the pH to 8.3.

B = ml of H₂SO₄ required to bring the pH to 4.5.

N = Normality of H₂SO₄ used.

Once the phenolphthalein and total alkalinities are determined then 3 types of alkalinities that is hydroxide, carbonate and bicarbonates are easily calculated from the following

| Values of P and T | OH | CO ₃ | HCO ₃ |
|-------------------|------|-----------------|------------------|
| P=0 | 0 | 0 | T |
| P<1/2 T | 0 | 2P | T-2P |
| P=1/2T | 0 | 2P | 0 |
| P>1/2T | 2P-T | 2(T-P) | 0 |
| P =T | T | 0 | 0 |

OBSERVATIONS:

| Sample details | Initial Reading (A) | Final Reading(B) | Reading (ml) | Alkalinity(mg) |
|----------------|------------------------|---------------------|--------------|----------------|
| Tap Water | | | | |
| | | | | |
| | | | | |
| Sewage | | | | |
| | | | | |
| | | | | |
| | | | | |

CALCULATION:

$$\text{Alkalinity in mg per Liter as CaCO}_3 = \frac{(A/B) \times N \times 1000}{\text{ml of sample}}$$

$$P = \text{Alkalinity mg/lit as CaCO}_3 = \frac{A \times N \times 1000}{\text{ml of sample}}$$

$$T = \text{Alkalinity mg/lit as CaCO}_3 = \frac{B \times N \times 1000}{\text{ml of sample}}$$

RESULT :

The alkalinity obtained for given sample of

Tap water =

Sewage =

CONCLUSION:

Large amount of alkalinity imparts a bitter taste to water. The main problem of alkaline water is the reaction that can occur between alkalinity and certain cations in waters. The resultant precipitation can cause damage to the water distribution system. Alkalinity is a major item that must be considered in calculating the lime and soda ash requirements in softening of water by precipitation methods. Water having an alkalinity content of less than 250 mg/l is desirable for domestic consumption.

Experiment No.7

Determination of pH

AIM:

To determine the pH value of given sewage sample and tap water.

THEORY:

pH is a term used to express the intensity of the acid or alkaline condition of a solution. It is a way of expressing the hydrogen ion concentration. The pure water consists of H⁺ ions combined with OH⁻ ions. But the process of dissociation takes place in pure water and hence, it contains some uncombined positively charged H⁺ ions. The water becomes acidic when positively charged H⁺ ions are in excess than negatively charged OH⁻ ions and it becomes alkaline when reverse is the case. For neutral waters concentration of H⁺ ions and OH⁻ ions are equal. The pH value is one of the important influencing parameter in treatment of water and waste water. A number of electrodes have been suggested for the electronic method of pH measurement. The glass electrode is standard electrode.

APPARATUS:

1. Standard pH meter.

PROCEDURE :

1. Standardize the pH metre by immersing the electrode in buffer solution of known pH.
2. Read the pH and correctly adjust with the control unit until the meter needle indicates the correct value of pH buffer solution.
3. Rinse the electrode in distilled water and then immerse in the sample. Let the needle settle and it indicates the pH.
4. Note the pH reading.

RESULT:

pH of sewage sample =

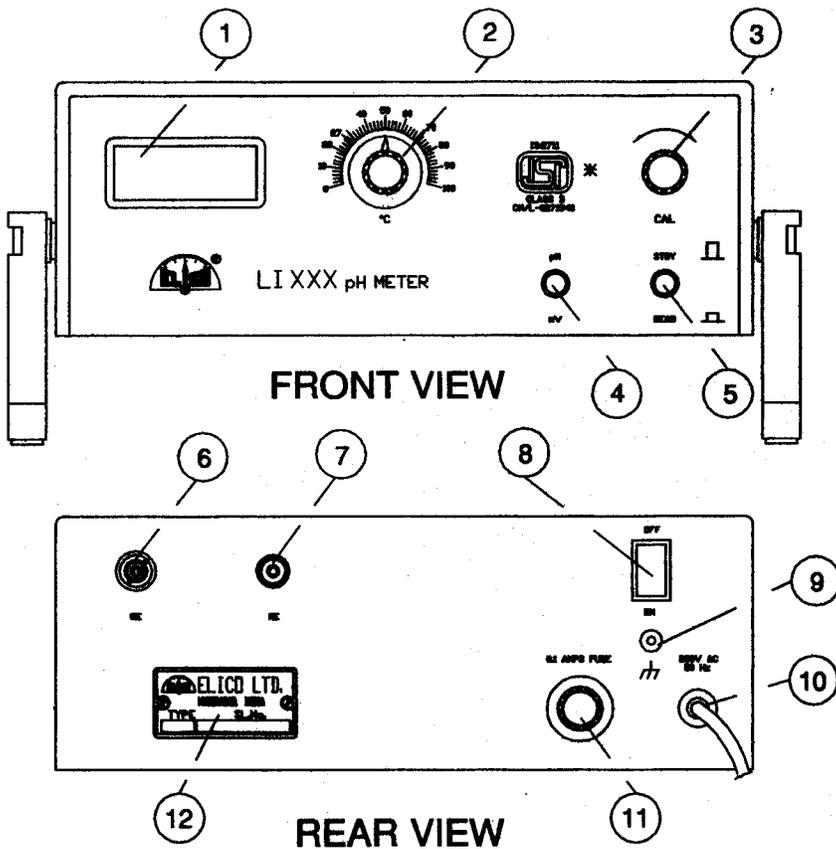
pH of tap water =

CONCLUSION:

It is desirable to maintain pH 6.5 to 8.5. The acidic water causes tuberculation and corrosion, where as high values of pH (>8.5) may produce incrustation, sediment

deposits, difficulty in chlorination and scale formation in water heating apparatus. In the field of water supplies pH is a factor that must be considered in chemical coagulation, disinfection, water softening and corrosion control.

pH METERS
LI 120 / LI 610



LEGEND

- | | |
|-------------------------------|---------------------------------|
| 1. 3 ½ Digit LED Display | 7. Reference Electrode Terminal |
| 2. Temperature Comp. Control | 8. Power ON/OFF Switch |
| 3. Calibration Control | 9. Ground Terminal |
| 4. pH/mV Selector | 10. Conductor Power cord |
| 5. STBY/READ Selector | 11. Fuse Holder with Fuse |
| 6. Glass Electrode Receptacle | 12. Number Plate |

* Applicable to LI 610 Model only
XXX represents the respective models either 120 or 610

Experiment No.8

Determination of Calcium, Magnesium & Total Hardness

AIM:

To determine the total hardness of the given sample of water.

THEORY:

Water hardness is the traditional measure of the capacity of water to react with soap, hard water requires a considerable amount of soap to produce lather. Scaling of hot water pipes, boilers and other house hold appliances is due to hard water. Hardness of water is not a specific constituent but is available and complex mixture of cations and anions. It is caused by dissolved polyvalent metallic ions. In fresh water principle hardness causing hardness ions are Ca, Mg. However, the ions Strontium, Iron, Barium and Manganese also contribute to hardness. The degree of hardness of drinking water has been classified in terms of equivalent CaCO_3 content as follows

| | |
|--------------------|-------------------------|
| Degree of hardness | mg/l as CaCO_3 |
| Soft water | 0 to 75 mg/lit |
| Medium water | 75 to 150 mg/lit |
| Hard water | 150 to 300 mg/lit |
| Very hard water | More than 300 mg/lit |

These cations plus the most important anions with which they are associated are shown below:

Cations causing Hardness



Anions

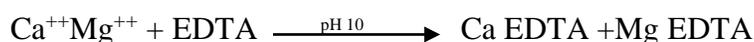


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Although the hardness is caused by cation it may be discussed in terms of carbonates (temporary) and non carbonate (permanent hardness). Carbonate hardness refers to the amount of carbonate and bicarbonates in the solution that can be removed or precipitation by boiling. This type of hardness is responsible for the deposition of scale in hot water pipes and kettles on carbonate hardness is caused by association of the hardness causing cation with sulphate, chloride or nitrate and is referred to as "Permanent hardness" it cannot be removed by boiling.

Public acceptability of the degree of hardness may vary considerably from community to community depending upon the local conditions and the associated anion. The threshold for magnesium is probably less than that of calcium.

In alkaline condition E.D.T.A reacts with Ca and Mg to form a soluble chelated complex. Ca and Mg ions develop wine red colour with Erichrome black-T indicator under alkaline conditions. When E.D.T.A is added as a titrant Ca and Mg bivalent ions get complexed resulting in the change of colour from wine red to blue which indicates the end point of the titration. The pH for this titration has to be maintained at 10.0 ± 0.1 . At a higher pH that is at about 12.0 Mg^{++} ion precipitates and only Ca^{++} get complexed resulting in a change from pink to purple which indicates the end pH of reaction..



APPARATUS AND REAGENTS:

- 1 Burette.
- 2 Pipette.
- 3 Conical flask.
- 4 Buffer solution
- 5 Inhibitor.
- 6 Erichrome black-T indicator.
- 7 Murexide indicator.
- 8 Sodium hydroxide 2N.
- 9 Standard E.D.T.A solution of 0.01N.
- 10 Standard Calcium solution.

PROCEDURE :

1. Take 50 ml of well mixed sample in a conical flask. Add 1-2ml of buffer solution followed by 1ml inhibitor
2. Add a pinch of Erichrome black T and titrate with standard E.D.T.A(0.01N) till urine-red colour changes to blue .Note down the volume of E.D.T.A required (A).
3. Run a reagent blank. Note the volume of E.D.T.A required (B).
4. Calculate the volume of E.D.T.A solution by $C = (A-B)$.
5. Calculate the total hardness as follows

$$\text{Total hardness as CaCO}_3 = \frac{C \times D \times 1000}{\text{ml of sample}}$$

Where C= volume of E.D.T.A required by the sample.

D = (Magnesium) CaCO₃ equivalent to 1.0 ml of E.D.T.A titrant

PROCEDURE:

1. Take 50 ml of well mixed sample in a conical flask.
2. Add 1ml of NaOH solution to raise the pH to 12 and a pinch of Murexide Indicator.
3. Titrate immediately with standard EDTA (0.01M) till pink colour changes to purple. Note down the vol. Of EDTA required. (A)
4. Run a reagent blank. Note the volume of EDTA required (B) and keep it a side to compare end points of sample titrations.
5. Standardize the EDTA (0.1m) solution following the procedure of CALCIUM HARDNESS from 1-4. Using standard calcium solution.
6. Calculate the **calcium hardness** as follows calcium Hardness as CaCO₃

$$= \frac{(A' \times D \times 1000)}{\text{ml of sample}}$$

where A' = volume of EDTA required by the sample

D = mg CaCO₃ equivalent to 1.0 ml of EDTA titrant.

Magnesium Hardness as CaCO₃ = Total Hardness as CaCO₃ – Calcium Hardness as CaCO₃

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OBSERVATIONS:

Total Hardness:

| Sl.No. | Sample | Initial reading | Final reading | Difference |
|--------|-----------|-----------------|---------------|------------|
| 1 | Tap water | | | |
| | | | | |
| | | | | |
| 2 | Sewage | | | |
| | | | | |
| | | | | |

Permanent Hardness

| Sl.No. | Sample | Initial reading | Final reading | Difference | Average |
|--------|-----------|-----------------|---------------|------------|---------|
| 1 | Tap water | | | | |
| | | | | | |
| | | | | | |
| 2 | Sewage | | | | |
| | | | | | |
| | | | | | |

CALCULATIONS:

$$\text{Hardness mg/lit} = \frac{A \times 1000 \times N}{\text{ml of sample.}}$$

$$\text{Temporary Hardness} = \text{Total hardness} - \text{Permanent hardness}$$

$$\text{Magnesium hardness} = \text{total hardness} - \text{Calcium Hardness}$$

RESULT:

Hardness mg/lit = .

Temporary Hardness =

Magnesium hardness =

CONCLUSION:

Scales formed act as insulations and cause enormous loss of fuel in boilers. It makes food tasteless. Hardness of water is important in determining the suitability of water for domestic and industrial uses.

Experiment No.9

Determination of Dissolved oxygen concentration.

AIM:

To determine the concentration of dissolved oxygen in the given sample.

THEORY:

Oxygen dissolved in water, often referred to as DO, is a very important parameter of water quality and is an index of physical and biological processes going on in water. There are two main sources of dissolved oxygen in water, i.e. diffusion from air and photosynthetic activity within water. Diffusion of oxygen from air to water is a physical phenomenon and depends upon the solubility of oxygen which, in turn, is influenced by factors like temperature, water movements, and salinity etc. Photosynthetic activity is a biological phenomenon carried out by autotrophs (mainly phytoplankton in water) and depends upon autotroph population, light conditions and available gasses etc. Non-polluted surface waters are normally saturated with dissolved oxygen, while presence of oxygen demanding pollutants (like organic wastes) causes rapid depletion of dissolved oxygen from water. Oxydizable inorganic substance, like hydrogen sulphides, ammonia, nitrites, ferrous iron etc. also cause decrease in dissolved oxygen. Eutrophic waters have a wide range of dissolved oxygen content while oligotrophic ones have a narrow range. Eutrophic bodies of water show a clinograde oxygen curve in which dissolved oxygen is more at surface and depletes fast with the depth, while oligotrophic bodies of water show an retrograde oxygen curve in which dissolved oxygen increases with depth. Oxygen is considered to be an A-1 limiting factor, especially in lakes and in waters with heavy load of organic material. Organisms have specific oxygen requirements, for example, 2-5 mg/l for most of the fishes. Low dissolved oxygen may prove lethal for many of the organisms.

The solubility of atmospheric oxygen in fresh water ranges from 14.6 mg/ lt. at 0⁰ C to about 7.0 mg/ lt. at 35⁰ C under one atmospheric pressure. Since oxygen is a poorly soluble gas, its solubility directly varies with the atmospheric pressure at any given temperature.

Analysis of DO is a key test in sanitary engineering practice. The following illustrations reveal importance of DO as a parameter:

- a) It is necessary to know DO levels to assess quality of raw water and to keep a check on stream pollution.
- b) In liquid waste, DO is the factor that determines whether the biological changes are brought out by aerobic or anaerobic organisms.
- c) DO test is the basis of BOD test which is an important parameter to evaluate organic pollution potential of wastes.
- d) DO is necessary for all aerobic biological wastewater treatment processes.
- e) Oxygen is an important factor in corrosion. DO test is used to control amount of oxygen in boiler feed waters either by chemical or physical methods.
- f) DO is necessary for all aerobic biological wastewater treatment processes.

APPARATUS AND REAGENTS :

1. BOD bottles (100-300ml).
2. Laboratory glass wares
3. Sodium thiosulphate solution (.025N)
4. Manganous sulphate solution
5. Alkaline potassium iodide solution
6. Starch indicator.
7. Sulphuric acid concentrated.

PROCEDURE:

Take a glass stopper BOD bottles of known volume and fill it with sample avoiding any bubbling. No air should be trapped in the bottle after the stopper is placed. Open the bottle and pour in it 1 ml of each Manganous sulphate solution and Alkaline potassium iodide solution using separate pipettes. If the volume of the sample is more than 200 ml then add 2 ml of the reagents instead of 1 ml. a precipitate will appear. Place the stopper and shake the bottle thoroughly. Add 2ml of conc. H_2SO_4 and shake thoroughly to dissolve the precipitate. Transfer whole content or 203 ml of the content in a conical flask. Add a few drops of Starch indicator. Titrate this against sodium thiosulphate solution and note the end point when initial blue colour turns to colourless

OBSERVATIONS:

| Sample Details | Titrant Consumed | | |
|----------------|----------------------|--------------------|-----------------|
| | Initial Reading (ml) | Final Reading (ml) | Net amount (ml) |
| 1) Tap Water | | | |
| i. | | | |
| ii. | | | |
| iii. | | | |
| 2) Sewage | | | |
| i. | | | |
| ii. | | | |
| iii. | | | |

CALCULATIONS

i) If 203ml of the sample is used for titration

DO of the sample = Volume of 0.025 N Sodium Thiosulphate Solution used.

(as 1ml of 0.025 N Na₂S₂O₃ consumed = 1 mg of DO)

ii) If a fraction of the content is used for titration

$$DO \text{ (mg/l)} = \frac{[V_1 \times N \times 8 \times 1000]}{[V_4 (V_2 - V_3) / V_2]}$$

where V₁ = Volume of titrant

 N = Normality of titrant

 V₂ = Volume of sampling bottle

 V₃ = Volume of Manganous sulphate solution and Alkaline potassium Iodide solution

 V₄ = Volume of fraction of content used for Titration.

CHEMICAL EQUATIONS:



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3. $\text{MnO}(\text{OH})_2 + 2 \text{H}_2\text{SO}_4 \longrightarrow \text{Mn}(\text{SO}_4)_2 + 3 \text{H}_2\text{O}$
4. $\text{Mn}(\text{SO}_4)_2 + 2 \text{KI} \longrightarrow \text{MnSO}_4 + \text{K}_2\text{SO}_4 + \text{I}_2$
5. $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}_2 \longrightarrow \text{Na}_2\text{S}_4\text{O}_6 + 2 \text{NaI} + 10 \text{H}_2\text{O}$
6. $2 \text{NaN}_3 + \text{H}_2\text{SO}_4 \longrightarrow 2 \text{HN}_3 + \text{Na}_2\text{SO}_4$
7. $\text{HNO}_2 + \text{HN}_3 \longrightarrow \text{N}_2 + \text{N}_2\text{O} + \text{H}_2\text{O}$

RESULT:

The dissolved oxygen content of tap water is found to be=

The dissolved oxygen content of sewage is found to be=

CONCLUSION:

Oxygen is an important factor in corrosion. DO test is used to control amount of oxygen

DO is necessary for all aerobic biological wastewater treatment processes .

Experiment No.10

Determination of BOD (Biochemical Oxygen Demand) of the sample

AIM:

To determine the BOD of the given sample

THEORY:

The rate of removal (i.e. consumption) of oxygen by micro organisms in aerobic degradation of the dissolved or even particulate organic matter in water is called as the BOD and it is used as an index of organic matter present in water, more the amount of oxygen required to degrade it biologically, hence more the BOD.

BOD is evaluated by measuring oxygen concentration in sample before and after incubation at 27° C for 3 days. Preliminary dilution and aeration of sample are usually necessary to ensure that not all the oxygen is consumed during the incubation period.

APPARATUS AND REAGENTS:

1. BOD bottles (100-300 ml)
2. Laboratory glass wares
3. Sodium Thiosulphate solution (0.025N)
4. Manganese sulphate solution
5. Alkaline potassium iodide solutions
6. Starch indicator
7. Sulphuric acid concentrated
8. Phosphate buffer solution
9. Magnesium sulphate solution
10. Calcium chloride solution
11. Ferric chloride solution
12. Sodium Hydroxide solution
13. Allylthiourea solution

PROCEDURE:

To prepare dilution water, aerate the BOD free distilled water in a glass container for about 1 hour. Add per liter of this water 1ml each of phosphate buffer solution, magnesium sulphate solution, calcium chloride solution Ferric chloride solution.

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Adjust the pH of sample to neutrality using 1N sulphuric acid or 1N sodium hydroxide solution as the case may be. Fill two sets of BOD bottle and add 1ml of Allylthiourea solution to each bottle. Determine the DO in one set immediately following the wrinklers method. Incubate the other set of BOD bottles at 27° C for 3 days and determine the DO after three days.

OBSERVATIONS:

| Sample details | DO immediately | | | DO after 5 days | | |
|----------------|----------------|-------|---------|-----------------|-------|---------|
| | initial | final | reading | initial | final | reading |
| Tap water | | | | | | |
| 1) | | | | | | |
| 2) | | | | | | |
| 3) | | | | | | |

CALCULATION:

$$BOD_3 @ 27^{\circ}C = (DO_I - DO_3) \times DF$$

DO_I = Initial Dissolved Oxygen in ml. before incubation.

DO₃ = Final Dissolved Oxygen in ml. after 3 days of incubation.

DF = Dilution factor.

RESULTS:

DF = Dilution factor.

$$BOD_3 @ 27^{\circ}C =$$

CONCLUSION:

The BOD of the given sample of waste-water is found out to be:
Based on the results the waste can be classified as (strong/ very strong/ moderately polluted waste).

Experiment No.11

Determination of COD (Chemical Oxygen Demand).

AIM:

To determine the chemical oxygen demand of the given sample.

THEORY:

Chemical Oxygen Demand is the measure of oxygen required in oxidizing the organic compounds present in water by means of chemical reactions involving oxidizing substance such potassium dichromate and potassium permanganate. Potassium dichromate is the most suitable oxidant but for waters having more than 2g/l of chlorides potassium permanganate is used, though the results are more variable because latter is self oxidizing.

The estimation of COD is of great importance for waters having unfavorable conditions for the growth of micro organisms, such as presence of toxic chemicals. In such waters BOD cannot be determined accurately. However, COD too is not a perfect index of organic compounds present in water because in this reaction, many inorganic compounds are also oxidized, and at the same time organic compounds remain unaffected.

APPARATUS AND REAGENTS:

1. COD reflux unit consisting of flat bottom flask with ground glass mouth and Liebig condenser, hot water bath or heating mantle.
2. Potassium dichromate solution.(0.025N)
3. Silver Sulphate powder.
4. Mercuric Sulphate.
5. Conc. Sulphuric Acid.
6. Ferroin indicator solution.
7. Ferrous ammonium Sulphate solution.(0.1N)

PROCEDURE:

Standardization: To standardize this solution, dilute 25ml of potassium dichromate solution to about 250ml with distilled water, add 20ml of sulphuric acid and cool it. Add

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5-6 drops of Ferroin indicator solution and titrate against ferrous ammonium Sulphate solution. The colour changes from blue green to a reddish blue at end point.

1. Take 20ml of sample in the flask of reflux unit.
2. Add 10ml of Potassium dichromate solution and pinch of each Silver Sulphate and Mercuric Sulphate
3. Add 30ml of sulphuric acid.
4. Attach Liebig condenser to the mouth of flask and heat the flask on a hot water bath or heating mantle for at least 2 hours to reflux the contents.
5. Cool the flask, detach from the unit and dilute its contents to about 150ml by adding distilled water.
6. Add 2-3 drops of Ferroin indicator solution and titrate against ferrous ammonium Sulphate solution. At the end point blue green colour of contents changes to reddish blue.
7. Repeat the same procedure for distilled water blanks.

OBSERVATION:

| | Blank | | | Sample | | |
|------|---------|-------|---------|---------|-------|---------|
| | Initial | Final | reading | Initial | Final | reading |
| i. | | | | | | |
| ii. | | | | | | |
| iii. | | | | | | |

CALCULATION:

$$\text{COD (mg/liter)} = \frac{[(B - A) \times N \times 1000 \times 8]}{V}$$

B: Volume of titrant used against blank.

A: volume of titrant used against sample.

N: Normality of sample.

V: volume of sample in ml.

RESULTS:

The chemical oxygen demand of the given sample =

CONCLUSION:

Maximum limit for COD is 250 mg/lit.

Experiment No.12

Determination of optimum Alum dosage (Jar test)

AIM:

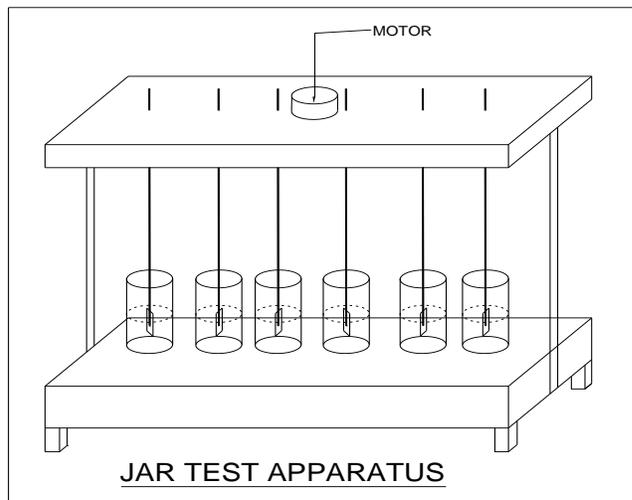
To determine the approximate optimum quantity of alum.

THEORY:

It was pointed out earlier that the best coagulant dose is determined in the laboratory and then adjusted by actual observations at the treatment plant. The common test which is performed to determine this approximate optimum quantity of coagulant is known as jar test. The amount of coagulant in the jar which produces a good floc with least amount of coagulant indicates the optimum dosage.

APPARATUS AND REAGENTS:

- 1) Glass beakers of capacity 1000ml.
- 2) Measuring jar.
- 3) Digital Weighing machine.
- 4) Jar testing apparatus.
- 5) Standard pH meter.
- 6) Alum
- 7) Hydrochloric acid.
- 8) Sodiumhydroxide



PROCEDURE :

- 1) Take about 6 beakers of 1000 ml capacity first
- 2) Take 200 ml of sample in one beaker. Adjust its pH to 6 by adding diluted hydrochloric acid
- 3) Keep it in the jar testing machine and start adding the alum in increment of 0.5gms stirring is started by the paddles connected to driving shaft through stirring rods placed inside jar. For first one minute rapid stirring is done and 3 minutes of slow stirring is done.
- 4) Observe the floc formation by adding 0.5 mg of alum and the process is repeated as in step 3 until the maximum floc formation occurs at the increment of 0.5 mg. Note down the amount of alum.
- 5) Now take 500ml of sample solution in each of the six jars .Set the pH with ranging from 5.5, 6.5, 7.5, 8.5, 9.5 and 10.5. Add the alum obtained in step 4 to all the jars .Start stirring with rapid mixing for 12 minutes. Note down the pH which gives maximum flocs. This pH is optimum pH.
- 6) Now set the pH obtained in step 5 in all six jars .Add the alum in increasing order from 0.5gm, 1.0gm, 1.5gm, 2.0gm, 2.5gm, and 3.0gm. Now note down the alum quantity which is optimum alum dosage. This gives the approximate optimum alum quantity of coagulant.

OBSERVATIONS:

| Jar No. | Dosage of Coagulant | Residual Turbidity NTU | pH |
|---------|---------------------|---------------------------|----|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

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Optimum alum content = $\frac{\text{Alum} \times \text{concentration of 1ml Alum} \times 1000}{\text{ml of sample}}$

RESULT:

The optimum pH obtained is=

The optimum alum content is=

CONCLUSION:

As per 10500 the turbidity for domestic water the permissible limit is 5JTU. It is observed that certain quantity of alum combines with colloidal particles to form floc which settles at the bottom

Experiment No.13

Determination of Turbidity

AIM:

To determine the concentration of Turbidity in the given sample.

THEORY:

The term turbid is applied to waters containing suspended matters that interfere with the passage of light. The turbidity may be caused by a wide variety of suspended matter which range in size from colloidal to coarse, dispersions, depending upon the degree of turbulence. Turbidity in natural waters is caused by silt clay organic matter, phytoplankton and other microscopic organisms. When light is passed through a sample having suspended particles, some of light is scattered by the particles. The scattering of the light is generally proportional to the turbidity. The turbidity of the sample is thus measured from the amount of light scattered by the sample taking a reference with standard turbidity suspension.

APPARATUS AND REAGENTS:

- 1 Turbidity meter
- 2 Sample tubes.

PROCEDURE:

Standard Hydrazine solution is required to calibrate the instruments. The standard method to prepare solutions is given below.

Solution I

Dissolve 1.0 gm of Hydrazine sulphate ($\text{NH}_2\text{NH}_2\text{H}_2\text{SO}_4$) in 100 ml of distilled water.

Solution II

Dissolve 10.00 gms of Hexamine (Hexa methylene tetra mine ($(\text{CH}_2)_6\text{N}_4$) in 100 ml of distilled water. Mix solution I&II. This solution is mixed thoroughly and allowed to stand for 24 hours at $25^\circ\text{C} \pm 3^\circ\text{C}$. This solution corresponds to 400 NTU. This solution is stable for about 2 to 3 months if kept in brown glass bottle and stored at around 25°C .

1. Take 25 ml of this solution (4000NTU) and add 75ml of double distilled water, 1000JTU solution can be prepared.

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2. Similarly other range of solution can be prepared by the successive dilution method.
3. Connect the mains lead to the mains supply $230V \pm 10\%$ 50Hz. Bring the power ON switch to ON position.
4. Allow the instrument to warm up for 15 minutes.
5. Keep the distilled water sample in the sample holder.
6. Adjust zero knob to get zero value on the display.
7. Keep standard solution as per the requirements (say 500 NTU solution) in the test tube holder and adjust the display to read the same value with CAL knob. Again put the distilled water sample in the sample holder and repeat zero adjustment procedure.
8. In case there is any minor variation readjust the display to zero. Now the instrument is calibrated.
9. The unknown solution is kept in the sample holder and the reading of turbidity is taken.

| Solution NTU | MI of 1000 NTU Solution | MI of distilled water |
|--------------|-------------------------|-----------------------|
| 900 | 90 | 10 |
| 800 | 80 | 20 |
| 700 | 70 | 30 |
| 600 | 60 | 40 |
| 500 | 50 | 50 |
| 400 | 40 | 60 |
| 300 | 30 | 70 |
| 200 | 20 | 80 |
| 100 | 10 | 90 |

OBSERVATIONS & RESULTS:-

Turbidity of given sample of water =

Turbidity of given sample of sewage =

CONCLUSION:-

Turbidity measurement is used to determine the effectiveness of the treatment produced with different chemicals and the dosages needed. Turbidity measurements help to gauge the amount of chemicals needed from day to day in operation of water treatment works.

Experiment No.14

Determination of percentage of Available Chlorine in Bleaching Powder.

AIM:

To determine the percentage of Available Chlorine in Bleaching Powder.

THEORY:

Chlorine is usually used for disinfection and decolourisation of water. The treatment by chlorination is cheap and reliable. The chlorine can be applied in water in one of the following ways

1. As bleaching Powder
2. As Chloramines
3. As free chlorine gas.

Bleaching powder (Calcium hypo chloride) when applied to water combines with hydrogen ions present in water to form hypochlorous acid HOCL. This process is called an hypochlorination.

The bleaching powder is a white powder. It contains 30 to 35% of active or available chlorine

APPARATUS AND REAGENTS:

1. Sodium thiosulphate (0.1N)
2. Acetic acid
3. Potassium iodide crystal
4. Bleaching Powder.
5. Conical flask.
6. Burette
7. Pipette
8. Measuring Cylinder

PROCEDURE:

Preparation of standard chlorine solution.

1. Take 2.5 gm of bleaching powder in a beaker .Add little quantity of distilled water and mix it properly make the volume to 250 ml by addition of distilled water.

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2. Take 25ml of standard chlorine solution and make the volume to 100 ml by addition of distilled water in a conical flask.
3. Add a pinch of potassium iodide crystals.
4. Add 10ml of acetic acid.
5. Keep the solution for 10 minutes as a contact period so that reaction takes place
6. After this add starch indicator to the solution so that blue colour is obtained.
7. Titrate the solution against standard Sodium thiosulphate of 0.1N till the colour changes from blue to colourless. Note down the volume of titrant.
8. Repeat the procedure for two or more samples and calculate the available chlorine in bleaching powder.

Available chlorine in bleaching powder % by weight

$$= \frac{(\text{Volume of titrant} \times \text{Normality} \times 35.45 \times 1000)}{(\text{Amount of bleaching powder taken})}$$

OBSERVATIONS:

| Sl No | Initial Reading (A) | Final Reading (B) | Difference in Reading (A-B) | Average Reading |
|-------|---------------------|-------------------|-----------------------------|-----------------|
| i | | | | |
| ii | | | | |
| iii | | | | |

CALCULATION:

$$\text{Available chlorine in mg/lit} = \frac{(\text{A-B}) \times \text{N} \times 35.45 \times 1000}{\text{ml of sample}}$$

$$\% \text{ of chlorine} = \frac{\text{Available chlorine in mg/lit} \times 1000}{100}$$

RESULTS:-

Available chlorine in mg/lit =

CONCLUSION:-

The bleaching powder contains 30 to 35% of active or available chlorine

Experiment No.15

Determination of Residual Chlorine

AIM:

To determine the residual chlorine in the given sample

THEORY:

Residual Chlorine is the Chlorine remaining in water after contact time (30 to 60 minutes) in chlorination process. The amount of chlorine that must be added to reach 0.1 to 0.2 mg/lit of residual content of non reacted chlorine in treated water. Under this requisition the dose of chlorine is equal to 0.5 to 2.0 mg/lit depending on impurities present in water. Chlorine may be applied in gaseous form (Cl₂) or in the form of bleaching powder (CaOCl₂). Chlorine combines with water to form hypochlorous and hypochloric acid .Hypochlorous acid dissociates to give OCl⁻ ions .



Quantity of OCl⁻ and HOCl⁻ depends upon pH of solution.Hypochloride also give OCl⁻ ions. HOCl rupture the cell membrane of microbes (the disease producing organisms).These also react with impurities like NH₃, oxidisable organic matter like ferrous ion, nitrates etc to form chlorinises and stable ion of latter respectively. In this way Cl⁻ is consumed by water sample.

APPARATUS AND REAGENTS:

- 1 Standard sodium thiosulphate solution.
2. Acetic acid.
3. Starch indicator.
4. Potassium Iodide crystal.
5. Bleaching Powder.
6. Conical flask.
7. Burette
8. Pipette
9. D.O.Bottle

10. Measuring Cylinder.

PROCEDURE:

1. Prepare standard solution by taking about 2.5 gm of bleaching powder and dilute it to 250 ml by adding distilled water.
2. Take about 25 ml of prepared solution.
3. Add a pinch of KI crystals to it and 10 ml acetic acid.
4. Add little amount of starch indicator.
5. Titrate against Standard Sodium thiosulphate till the colour changes from blue to colourless
6. Calculate the residual chlorine by using the formula

$$\text{Residual Chlorine} = \frac{(A-B) \times N \times 35.45 \times 1000}{\text{ml of sample}}$$

OBSERVATIONS:

| Sl No | Initial | Final | Reading | Average |
|-------|---------|-------|---------|---------|
| i | | | | |
| ii | | | | |
| iii | | | | |

$$\text{Residual Chlorine} = \frac{(A-B) \times N \times 35.45 \times 1000}{\text{ml of sample}}$$

RESULTS:-

Residual Chlorine in given sample =

CONCLUSION:

The acceptable value of chlorides are 200mg/lit

The chloride content in given sample of is found out to be =

Experiment No.16

Determination of Iron

AIM:

To determine the concentration of iron in the given sample

THEORY:

Iron is found in all natural water both in oxidized (ferric) and reduced (ferrous) forms. In anoxic reducing environment like ground water most of iron occurs in the ferrous state (as ferrous bicarbonate under alkaline condition) under aerobic conditions. Ferric hydroxide a brown insoluble substance. During oxidation of ferrous bicarbonate to ferric hydroxide carbon-di-oxide is released. Ferric iron is an important plant nutrient and considered to be quantitatively the most important trace metal for autotrophs because of its indispensability for many enzymes and red oxide process. Some microorganisms (like Leptothrix) are capable of utilizing dissolved iron (ferrous state) as an energy source and convert ferrous into ferric oxide.

APPARATUS AND REAGENTS:

1. Concentrated hydrochloric acid.
2. Hydroxylamine hydrochloride solution.
3. Ammonium acetate buffer solution.
4. Phenolathraline solution.
5. Standard iron solution.
6. Spectrophotometer
7. Conical flask.
8. Pipette.

PROCEDURE:

1. Take 6 samples of 50 ml each in a conical flask and add 2ml of hydrochloric acid and 1ml of hydroxylamine hydrochloride solution and iron solution (0.2, 0.4, 0.6, and 0.8) each in 5 flasks except blank solution.
2. Boil till content are reduced to half the original volume.
3. Cool and add 10 ml of ammonium acetate buffer solution and 2ml of Phenolathraline solution.

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4. Add distilled water to make the volume 100ml. Let it stand for 10 minutes and then record the absorbance on spectrophotometer of 530nm water as blank.
5. Pipette 1,2,4,6,8,12,14,16 ml of standard iron solution in Nessler's tubes and repeat steps 1 to 4 and record absorbance for each.
6. Plot a standard curve from these values and deduce the total iron content of sample in mg/lit by comparing the absorbance of the sample with standard curve.

OBSERVATIONS:

Wavelength used $\lambda = 530\text{nm}$.

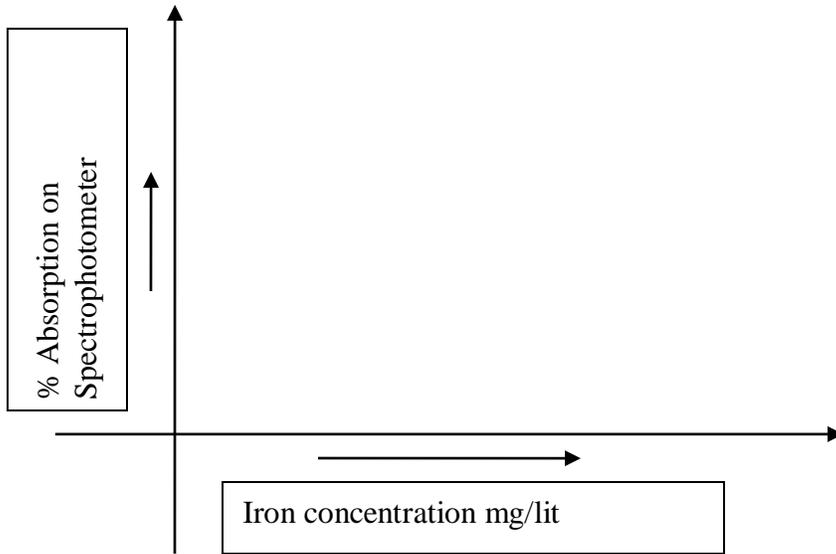
| Sl.No. | Concentration | Transmission |
|--------|---------------|--------------|
| i | | |
| ii | | |
| iii | | |
| iv | | |
| v | | |
| vi | | |

RESULTS:-

The concentration of iron in the given sample =

CONCLUSION:

Determination of iron is useful to select the treatment unit and design of treatment units. It is used to determine the efficiency of treatment units. The ratio of iron to manganese is a characteristic factor that determines the type of treatment unit used as well as the amount of organic matter present in water. It is also used to aid in the solution of problems in the distribution systems where iron fixing bacteria are trouble some. Iron determination is helpful in assessing the extent of corrosion and aiding in control of corrosion. The permissible limit for Iron is 0.3mg/lit



Experiment No.17

Determination of Fluorides

AIM:

To determine the Fluoride content in the given sample of water

THEORY:

Fluorides are more common in ground water than in surface water. The main source of fluoride in water are different fluoride bearing rocks. The maximum permissible limit of fluoride in drinking water is recommended to be 1.5 mg/lit by world Health Organization. When present in high concentration it causes mottling of teeth, Skeletal fluorosis forward bending of vertebral column, deformation of knee joints and other parts of body and even paralysis (Paraplegia and Quadriplegia). The test is based on the fact that fluoride ion combines with Zirconium ion to form a stable complex ion, ZrF_6 and this results in bleaching the reddish colour of Zirconium and Alizarin combination. The decrease in intensity of colour is directly proportional to fluoride concentration.

APPARATUS AND REAGENTS:

- a) Alizarin red solution
- b) Zirconyl acid solution
- c) Standard Fluoride solution.
- d) Spectrophotometer
- e) Conical flask

PROCEDURE:

1. Take 100 ml of sample in a conical flask.
2. Add 5ml of alizarin red solution and Zirconyl acid solution wait for one hour and then note absorbance on Spectrophotometer at 520nm.
3. Use distilled water as blank solution run the standard fluoride solution of various concentrations in similar manner and record the absorbance for each.
4. Plot a standard curve between concentration and absorbance of standard solution (Fluoride solution) of various concentrations in similar manner.
5. Find out fluoride content of the sample by confirming its absorbance with standard curve and express the result as mg/lit

OBSERVATIONS:

Wavelength used $\lambda = 520\text{nm}$.

Concentration of standard fluoride solution = 10mg/lit

| Sl.No | Concentration in mg/lit | ml of sample added | % absorbance reading on Spectrophotometer |
|-------|-------------------------|--------------------|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| | Blank | | |

RESULTS:-

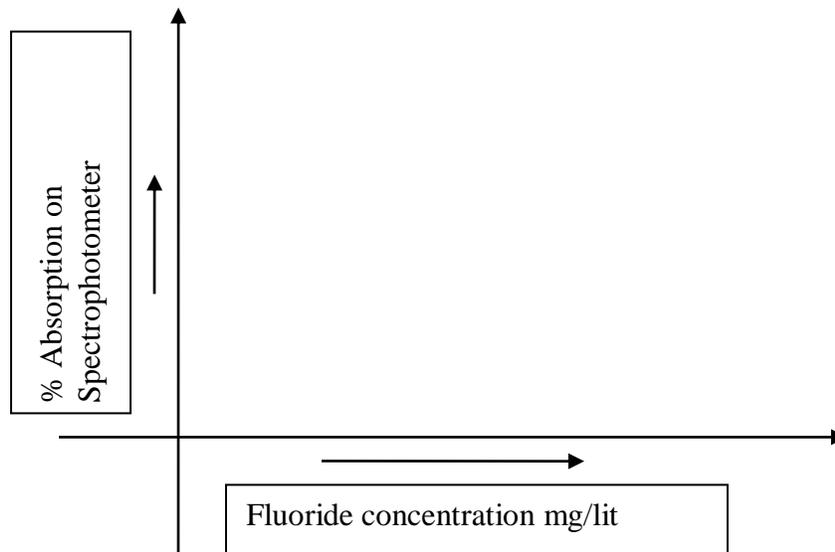
The fluoride content in the given sample of water =

CONCLUSION:

The acceptable value of fluoride is 1mg/lit. Presence of large amounts of fluoride is associated with dental and skeletal fluorosis and inadequate amounts with dental caries.

The Fluoride content in given sample of is found out to be =

Graph of Fluoride concentration Vs % Absorption on Spectrophotometer



Experiment No.18

Determination of Nitrate Nitrogen

AIM:

To determine the concentration of Nitrate Nitrogen in the given sample.

THEORY:

Nitrate is the highest oxidized form of nitrogen and in water its most important source is biological oxidation of nitrogenous organic matter of both autochthonous and all ochthonous origin. Domestic sewage and agricultural runoff are the chief sources of all ochthonous nitrogenous organic matter. Metabolic wastes of aquatic community and dead organisms add to the autochthonous nitrogenous organic matter. Nitrifying bacteria (ammonifying bacteria, Nitrosomonas, Nitrobacteria) play significant role in oxidation of such organic matter. Certain nitrogen fixing bacteria, i.e. azobacter and algae i.e. blue-greens – like anabaena, nostoc, have the capacity to fix molecular nitrogen in nitrates. In ground water nitrates may find way through leaching from soil and at times by contamination. The high concentration of nitrate in water is indicative of pollution. This is an important plant nutrient, when present in excess, it causes ubiquitous growth of algae, often present in blooms. High nitrate content i.e. more than 40 mg/ lt. may cause blue-baby disease. Significant sources of nitrate are chemical fertilizers, decayed vegetable and animal matter, domestic effluents, sewage sludge disposal on land, industrial discharge, leachates from refuse dumps and atmospheric washout. Nitrate reacts with phenol disulphonic acid produces a nitro-derivative which in alkaline medium develops a yellow colour. The colour produced follows Beer's law and is directly proportional to concentration of nitrate present in the sample.

APPARATUS AND REAGENTS :

1. Colorimeter/ spectrophotometer.
2. Nessler tubes, cap. 100 ml.
3. Beakers, cap. 100 ml.
4. Water bath.
5. Phenol disulphonic acid.
6. Std. nitrate solution.
7. Potassium Hydroxide solution (12N)

METHOD:

The method adopted here is the Phenol Disulphonic Acid (PDA) method.

PROCEDURE:

1. Take 25 ml of sample in a crucible and evaporate it to dryness on a hot water bath.
2. Add 0.5 ml of PDA to the residue and dissolve with the help of a glass rod. Add 5 ml of distilled water and 1.5 ml of potassium hydroxide solution.
3. Stir for thorough mixing. Take the supernatant of the yellow colour and read its absorbance/ transmittance on spectrophotometer at 410 nm.
4. Use the distilled water as blank.
5. Process the standard nitrate solutions in similar manner and note the transmittance for each.
6. Plot a standard curve between transmittance and concentrations of various standard solutions.
7. Deduce the nitrate nitrogen for the unknown sample from this curve, and express the result in mg/ lt.

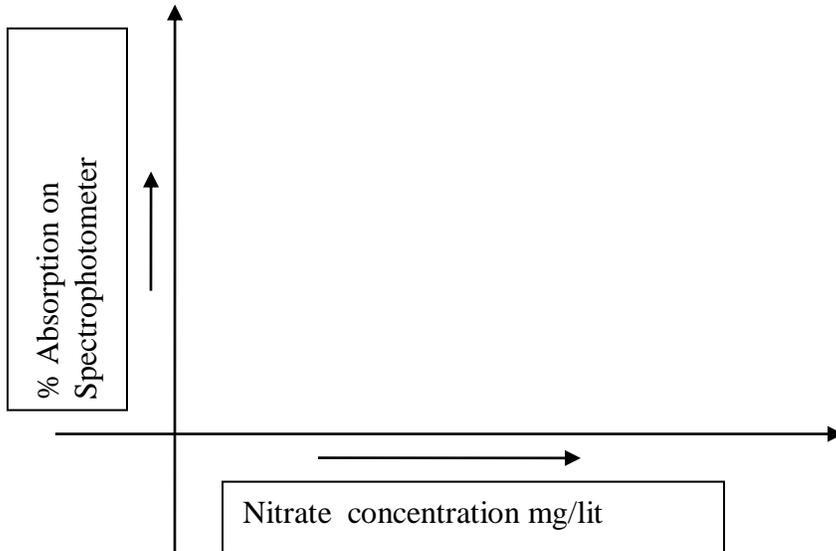
OBSERVATIONS:

| Concentration (mg/lt.) | % Transmission |
|------------------------|----------------|
| 0.0 | |
| 5.0 | |
| 10.0 | |
| 15.0 | |
| 20.0 | |
| 25.0 | |
| 30.0 | |
| 35.0 | |

RESULT:

The concentration of Nitrate Nitrogen in the given sample

CONCLUSION: It is used to assess the self purification properties of water bodies and nutrient balance in surface water and soil. It is useful to find out state of decomposition of organic matter present in waste waters.



Experiment No.19

Determination of Total Count

AIM:

To determine the Total count of the given sample

THEORY:

Many bacteria are found in water. In surface water the coli form group is the rod shaped non-pathogenic bacteria whose presence or absence in water indicates presence or absence of local pollution and hence of the pathogens. It is useful to assess the degree of contamination of receiving waters due to domestic sewage. It is useful to find efficiency of disinfectant and treatment units efficiency in pathogenic bacterial removal

APPARATUS AND REAGENTS:

1. Petri dish
2. Quebec Colony Counter
3. Pipette
4. Molten Nutrient Agar

PROCEDURE:

1. A suitable volume of water sample (or dilution of water sample with sterilized sample) is transferred to a sterilized Petri dish by means of sterile pipette.
2. Molten nutrient agar cooled at 45°C is added and thoroughly mixed with sample in the Petri dish.
3. When the mixture solidifies the Petri dish is transferred to the incubator at 37°C and left there for 24 hours incubation
4. The bacterial colonies which are formed are then counted with the help of magnifying glass.
5. The results of colony count are computed for 1 ml of water sample. For potable water it should not exceed 10/ml.

RESULT:

Total count of the given sample=

CONCLUSION:

Total count for potable water should not exceed 10/ml.

Experiment No.20

Determination of MPN

AIM:

To determine the Most Probable Number of the given sample

THEORY:

Many bacteria are found in water. In surface water the coli form group is the rod shaped non-pathogenic bacteria whose presence or absence in water indicates presence or absence of local pollution and hence of the pathogens .E-Coli is the predominant member of the fecal coli form group. It is a parasite living only in the human or animal intestines Detection of E-coli in drinking water is taken as evidence of recent pollution with human or animal feces. The intestinal tract of human beings contains countless rod shaped bacteria known as coli form organisms. Each person discharges 100 to 400 billion coli form organisms per day in addition to other kinds of bacteria. The presence of coli form organisms are taken as an indication that pathogenic organisms may also be present and the absence of coli form organism is taken as an indication that the water is free from disease producing organisms.

APPARATUS AND REAGENTS:

1. Durhamis tubes
2. MAC Conkey's Broth.
 - a. Peptone 2.4gm
 - b. NaCl 0.63gms
 - c. Bile Salt 0.63gms
 - d. Lactose 1.25gms

Transfer them to 250 ml flat bottom flask and dissolve in distilled water. Adjust pH at 7.4 Transfer the broth to 10 ml culture tubes, plug the mouth of the tubes with non absorbent cotton and sterilize in the autoclave.

PROCEDURE:

1. This method is based on statistics .The water sample of different dilution is fed to three rows of test tubes consisting five test tubes in each row containing sterilized Mac Conkey's broth.

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2. Five tubes are inoculated (mixed) with 10 ml of water sample in each tube, Another 5 test tubes are mixed with 1ml of water sample in each tube, and another 5 test tubes are inoculated with 0.1 ml of water sample in each tube.
3. The test tubes without being disturbing the rows are kept for incubation at 35°C for 24 hours .Coli form present in any test tube will react with the broth and will produce gas and the colour of the broth will be changed.
4. This will be considered as positive tubes and unchanged tubes are negative tubes. After 24 hours incubation the result obtained is presumptive test.
5. The tests showing +ve presumptive test are further subjected to confirmatory test, which will eliminate certain bacteria of non-sanitary importance. A portion from the fermentation tube (tube evolving gas) is transferred to a tube containing brilliant green bile broth. This is incubated for 48 hours, and if gas is formed it will be a positive confirmatory test for the presence of coli form bacteria.
6. After determining the positive test tubes, the most probable number (MPN) of the coli forms in the total water sample is determined.
7. Tables which permit quick determination of MPN/100 ml of total sample are readily available. Water quality is classified on the basis of coli form count as given below

| Groups | Quality | MPN/100 ml |
|-----------|----------------|------------|
| Class I | Excellent | 0 |
| Class II | Satisfactory | 1-3 |
| Class III | Suspicious | 4-10 |
| Class IV | Unsatisfactory | >10 |

OBSERVATIONS

| Sl.No | Proportion | No. of tests | Positive results in percent | Difference | Reciprocal of column (2) | Multiplication of columns (5) & (6) |
|-------|------------|--------------|-----------------------------|------------|--------------------------|-------------------------------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 |
| 1 | | | | | | |
| 2 | | | | | | |
| 3 | | | | | | |
| 4 | | | | | | |
| 5 | | | | | | |
| Total | | | | | | |

RESULT:

The Most Probable Number of the given sample=

CONCLUSION:

The purpose of this test is to estimate the number of coli forms in water sample as an index of the magnitude of biological contamination. E-coli determination is useful in the selection of water supplies for human needs .It is useful to assess the degree of contamination of receiving waters due to domestic sewage. It is useful to find efficiency of disinfectant and treatment units efficiency in pathogenic bacterial removal

Experiment No.21

Determination of Chlorine Demand By Break Point Chlorination

AIM:

To determine the Chlorine demand By Break point chlorination of the given sample

THEORY:

The Chlorine demand of water is the difference between the amount of chlorine applied and the amount of free, combined or total available chlorine remaining at the end of the contact period. Out of total amount of chlorine added only a relatively small part is spent for disinfection of water while the major part is consumed in reaction with organic matter and soluble iron and manganese in water.

Factors affecting chlorine demand

1. Form of chlorine.
2. pH
3. Concentration of impurities.
4. Type of Organism
5. Temperature of water.

APPARATUS AND REAGENTS:

1. Burette
2. Pipette
3. Conical Flask
4. Acetic acid
5. Potassium Iodide (KI) crystals.
6. Sodium Thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$)
7. Starch indicator

PROCEDURE:

1. Take six to eight glass beakers of 1000 ml capacity and add 1000ml water sample to each
2. Weigh accurately the amount of bleaching powder calculated equal to a dosage of 2, 4, 6, 8, 10, 12mg and add to each beaker.
3. Stir with glass rod to dissolve the powder completely.
4. Allow it for contact period of 15 to 30 minutes without any disturbances

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5. Take a suitable quantity of sample (200 ml) in a conical flask.
6. Add 1gm of KI crystals in one test tube 10% KI solution and 5 to 10 ml of acetic acid .Mix it well with a glass rod.
7. Titrate it with sodium thiosulphate solution to calculate the residual chlorine.

OBSERVATIONS:-

| Dosage ml | Quantity of bleaching powder in mg | Residual chlorine in mg/lit |
|-----------|------------------------------------|-----------------------------|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

CALCULATIONS:-

$$\text{Quantity of bleaching powder for required dose} = \frac{\text{Dosage} \times 100}{\text{Available chlorine in bleaching Powder}}$$
$$\text{Residual Chlorine (mg/lit)} = \frac{\text{B.R} \times \text{N} \times 35.45 \times 1000}{\text{Weight of sample taken}}$$

RESULT:

Residual Chlorine (mg/lit) =

CONCLUSION:

The treatment of water with chemicals to kill bacteria is termed as disinfection of water. Break point chlorination involves the addition of sufficient chlorine .So as to oxidize all the organic matter reducing substances and free ammonia in raw water leaving behind mainly free available chlorine which posses strong disinfection action against pathogen used for disinfection of water. Chlorine compounds are used in water and water treatment systems. Chlorination, ionization, ultraviolet rays method, excess lime process and applicant of iodine and Bromine method are the Principle method.

Viva Questions

1. What are the reasons for the analysis of water?
 2. Mention the precautions to be taken while collecting the sample of water to be analyzed.
 3. What is alkalinity? How is it determined?
 4. What are the facts to be noted in solving the problems on hardness?
 5. Differentiate
 - a) CO₂ acidity and Mineral acidity.
 - b) Dissolved solids and suspended solids.
 - c) Temporary hardness and Permanent Hardness
 - d) Palatable and potable water.
 - e) Taste and odour.
 6. Give reasons
 - a) The excess hardness of water is undesirable.
 - b) The water has lower content of salt than sewage
 7. Define BOD. What is its importance in sewage analysis?
 8. What are the limitations of BOD test?
 9. Define COD. Discuss how is it determined?
 10. What is the minimum amount of DO to be present in sewage sample before disposal?
 11. What is jar test? Explain
 12. What is hardness scale?
 13. What are the reagents used to determine the percentage of chlorine in bleaching powder?
 14. State the limits of chlorides content in tap water and sewage.
 15. What are the ill effects caused due to the presence of sulphates in water?
 16. What are the advantages of chlorination?
 17. Define MPN.
 18. What is break point chlorination?
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