

**MUFFAKHAM JAH COLLEGE OF
ENGINEERING AND TECHNOLOGY**

DEPARTMENT OF CIVIL ENGINEERING

**ENVIRONMENTAL ENGINEERING LABORATORY
MANUAL**

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ESTIMATION OF CHLORIDES IN THE SAMPLE WATER

AIM:- To determine the concentration of chlorides in the given sample water.

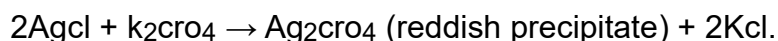
APPARATUS:- Glassware.

THEORY:- Chloride is a major inorganic constituent of natural waters. Chlorides ions may be in combination with cations like calcium, magnesium, iron and sodium. Chlorides of these minerals are present because of their high solubility in water. The other sources of chlorides in the water are intrusion of sea water into fresh water bodies, pollution of industries waste and domestic wastes.

The chloride content of water is measured by titrating the water sample with standard silver nitrate solution (AgNO_3) using potassium chromate $\text{K}_2\text{Cr}_2\text{O}_7$ as indicator. The silver first reacts with all chlorides thereby forming silver chlorides as indicated by the following equation.



The silver chloride so formed then reacts with potassium chromate (indicator) to form silver chromate producing reddish precipitate which indicates the end point. This reaction is indicated by the following reaction.



The amount of silver nitrate required to produce reddish precipitate determines the amount of chlorides present in the water sample.

RELEVANCE:- The permissible limit of chloride in domestic water supplies is upto 250ppm. Although chlorides in excess of 250ppm are not harmful, but they cause unpleasant taste to water, thus rendering the water unacceptable for drinking purpose. The presence of large quantity of chlorides in water indicated its pollution due to sewage. The chloride concentrations of raw waters being used for public water supplies should therefore tested regularly, as to detect any sudden increase in their chloride content and the possibility of the organic pollution of the water source.

REAGENTS:-

- 1. Potassium chromate indicator:-** Dissolve 10.0grams K_2CrO_4 in a little distilled water. Add silver nitrate solution until a red precipitate is formed. Let stand 12 hours, filter and dilute it to 200ml with distilled water.
- 2. Standard silver nitrate solution (0.0141N):-** Dissolve 2.395 grams AgNO_3 in distilled water and dilute it to 1000ml. standardize against 0.0141N sodium chloride solution as per the procedure described below. Store it in a brown glass bottle.
- 3. Standard sodium chloride (0.0141N):-** Dissolve 824.1mg NaCl in chloride free water and dilute it to 1000ml.

PROCEDURE:-

1. **Standardization of AgNO₃ (Titrant):-** Take 20ml standard sodium chloride solution and dilute it to 100ml. add 1ml K₂CrO₄ indicator. Titrate with AgNO₃ solution to pinkish yellow end point. Note down the volume of AgNO₃ consumed and determine the normality of AgNO₃ using the relation

$$N_1V_1 = N_2V_2$$

Where

N₁ = Normality of AgNO₃ (unknown)

N₂ = Normality of NaCl solution (0.0141)

V₁ = Volume of AgNO₃

V₂ = Volume of NaCl solution (20ml)

Adjust the normality to AgNO₃ to 0.0141N using the relation $N_1V_1 = N_2V_2$

2.

3. **Determination of chloride content in the sample water:** Take a suitable volume of sample water (say 50ml) in a conical flask and dilute it to 100ml. with distilled water. Add 1ml K₂CrO₄ indicator and titrate with 0.0141N AgNO₃ solution in a PH range of 7-10, to a pinkish yellow end point. Note down the volume of AgNO₃ consumed.

OBSERVATIONS:-

S.No	Burette Readings		Vol. of Titrant used
	Initial	Final	

CALCULATIONS:-

$$\text{Chloride content in mg/l} = \frac{\text{Vol of AgNO}_3 \times N \times 35450}{\text{ml of sample water}}$$

RESULTS:-

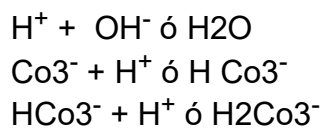
INTERPRETATION OF RESULTS:-

ALKALINITY

AIM:- To determine the alkalinity of the given sample water.

APPARATUS:- PH meter and Glassware.

THEORY:- The alkalinity of a water sample is its capacity to neutralize acids. Alkalinity is mainly due to presence of carbonate, bicarbonate, hydroxides and less frequently, borate's, silicates and phosphates. It is expressed in mg/l of calcium carbonates. Two types of alkalinities are generally calculated, namely, phenolphthalein alkalinity and methyorange alkalinity. Based on these two different forms of alkalinities can be estimated. Alkalinity measurements are made by titrating the sample water with an acid and determining the hydrogen equivalent. Hydrogen ions from the acid reacts with the alkalinity according to the following equations.



If acid is added slowly to the water and the PH it recorded for each addition, a titration curve is obtained as shown below.

Graph

The significant aspects of the curve are the inflection points that occur at approximately PH8.3 and PH4.5. At PH8.3, all hydroxides and half of the carbonates are neutralized. At PH4.5, remaining half of the carbonates and bicarbonates are neutralized. Thus the amount of acid required to titrate a sample to PH4.5 is equivalent to the total alkalinity of the sample water.

RELEVANCE:- Alkalinity plays a very important role in chemical coagulation and in biological waste treatment processes, especially in aerobic digestion as this provides buffering systems. Excess alkalinity imparts a bitter taste to waste. Alkalinity is an important parameter in evaluating the optimum coagulant dosage. The permissible limit of carbonate alkalinity in domestic water supplies is 120ppm.

CHEMICAL REAGENTS:-

- i) Standard sulphuric acid (0.02N)
- ii) Phenolphthalein indicator
- iii) Methyl orange indicator
- iv) Sodium carbonate solution.

PROCEDURE 1:-

1. **Standardization of H₂SO₄ (titrant) by potentiometric titration**:- Take 40ml Na₂CO₃ solution and add 100ml distilled water. Titrate with H₂SO₄ till PH of 1-2 is reached. Add acid in amounts of 0.5ml and note down the corresponding PH. Plot a graph between volume of H₂SO₄ and the corresponding PH. From the graph, determine the volume of H₂SO₄ at a PH of 4.5. Calculate the normality of the acid using the formula.

$$N = \frac{53 \cdot C}{A \cdot B}$$

Where

A = Volume of Na₂CO₃ (40ml)

B = Grams of Na₂CO₃ in 1 lit. = 2.5 grams

C = Volume of H₂SO₄ for a PH of 4.5

Adjust the normality of H₂SO₄ to 0.02N using the relation

$$N_1V_1 = N_2V_2$$

2. **Determination of 'P' and 'T' alkalinity**:- Take 25ml sample water and make it to 50ml using distilled water. Add 2 drops of phenolphthalein indicator and titrate with 0.02N H₂SO₄ till pink colour disappears. Note down the volume of H₂SO₄ as V₁. To the same sample add 2 drops of methylorange indicator and titrate with H₂SO₄ till colour changes to pinkish yellow. Note down the total volume of acid consumed as V.

OBSERVATIONS:-

S.No	Burette Readings		Vol. of Titrant used
	Initial	Final	

Volume of acid	PH

PROCEDURE 2 :-

.Take 50 ml of sample water in a conical flask and add 3 to 4 drops of phenolphthalein indicator, color changes to pink.

.Titrate against 0.02 N HCL till colorless as end point.

. Note down the volume consumed from burette to measure phenolphthalein alkalinity. .To the same sample add 3-4 drops of methyl orange indicator and color changes to orange.

. Titrate it against 0.02 N HCL till solution changes to pink as end point.

. Note down the volume consumed from burette to measure methyl orange alkalinity.

. The sum of phenolphthalein alkalinity and methyl orange alkalinity gives total alkalinity.

Calculations:- Total alkalinity = $\frac{V1 \times 1000}{\text{ml of water sample}}$ + $\frac{V2 \times 1000}{\text{ml of water sample}}$

where V1 and V2 are volume consumed from burette for phenolphthalein alkalinity and methyl orange alkalinity.

CALCULATIONS for First Procedure:-

Phenolphthalein Alkalinity "P" = $\frac{V1 \times N \times 50000}{\text{ml of sample}}$
as mg/l CaCO_3

Total alkalinity "T" as mg/lit as CaCO_3 = $\frac{V \times N \times 50000}{\text{ml of sample}}$

Calculate the alkalinity due to various constituents using the table given below.

	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
If $P=0$	0	0	Total Alkalinity T
If $P=T/2$	0	2P	$T - 2P$
If $P<T/2$	0	2P	$T - 2P$
If $P>T/2$	$(2P - T)$	$2(T - P)$	0
If $P=T$	T	0	0

RESULT:-

GRAPH:- Plot a graph taking PH value on Y-axis and corresponding volume of acid on X-axis. From the graph obtain the volume of acid corresponding to a PH of 4.5. Also mark the inflection points (i.e. point on the graph corresponding to PH 8.3 and 4.5)

INTERPRETATION OF RESULTS:-

BIOCHEMICAL OXYGEN DEMAND (B.O.D.) TEST

AIM:- To determine the Biochemical oxygen demand of the sewage sample.

APPARATUS:-

- i) Glassware
- ii) B.O.D. bottle or incubation bottle
- iii) Incubator.

THEORY:-

The B.O.D. of sewage (domestic or industrial) or any polluted waste water is defined as the amount of oxygen required for the biochemical decomposition of dissolved organic solids to occur under aerobic conditions & under standardized time & temperature. The laboratory test for B.O.D is an empirical test conducted at a temperature of 20⁰C & an incubation period of 5 days (sometimes 7 days). This test is based on the principle of determination of dissolved oxygen before & after incubation for a specified period (5 days) at standard temperature (20⁰C). The difference in D.O value gives oxygen utilized by the bacteria for the oxidation of organic matter, which is expressed as mg/lit. The rate of oxidation depends upon the amount & type of organic matter, dissolved oxygen, temperature, types of organisms & environmental conditions. Under natural conditions, complete oxidation of organic matter takes about 2-3 months but within 10 days nearly 90% biological demand is satisfied after which the rate of oxygen consumption becomes very slow. In the laboratory, 5-days BOD is tested which gives 60-70% of total demand.

The decomposition or decay of organic substance takes place by the help of enzymes, which are produced by the secretion of protein molecules from the living organisms. The organic matter & some of the complex mineral portions of those substances are utilized as a source of energy by a series of living organisms. A series of biochemical reactions is thereby set in motion, & polluted waters are returned to a normal state or purity.

RELEVANCE:-

The BOD has tremendous applications in the field of sanitary engineering. Some of these applications are as follows.

- i) To measure the strength of sewage & wastes.
- ii) To determine the amount of pollution in a stream.
- iii) In the design of water treatment facilities.
- iv) To determine the efficiency of the treatment units.
- v) In the establishment of stream & effluent standards for stream pollution regulatory agencies.

THE NORMAL VALUES OF B.O.D FOR RAW & TREATED SEWAGE (DOMESTIC) ARE GIVEN BELOW.

Nature of sewage	BOD ₅ at 20 ⁰ C in mg/l
1, Strong sewage	450 – 500

2. Average sewage	350
3. Weak sewage	250
4. Std. filter sewage effluent	20
5. Very good filter sewage effluent	5 – 10

REAGENTS FOR DILUTION WATER:-

- i) Distilled water for dilution.
- ii) Phosphate buffer solution consisting of 8.5g KH_2PO_4 , 21.75g K_2HPO_4 , 33.4g $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$ and 1.7g of NH_4Cl dissolved in 1 lit. PH of this solution is 7.2.
- iii) Magnesium sulphate solution consisting of 22.5g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ dissolved in distilled water & diluted water diluted to 1 lit.
- iv) Calcium chloride solution consisting of 27.5g CaCl_2 dissolved in distilled water & diluted to 1 lit.
- v) Ferric chloride solution, consisting of 0.25g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ dissolved in distilled water & diluted to 1 lit.

REAGENT FOR D.O MEASUREMENT: (By AZIDE MODIFICATION METHOD)

- i) **Manganese sulphate solution**: Dissolve 480g $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ in distilled water and dilute it to 1 lit.
- ii) **Alkali – Iodide – Azide reagent**: Dissolve 500g NaOH and 150g KI in distilled water and dilute it to 1 lit. add 10g sodium Azide(NaN_3) dissolved in 40ml distilled water
- iii) **Concentrated H_2SO_4** .
- iv) **Starch**:- Dissolve 5g starch in 1 lit boiling distilled water.
- v) **Sodium tiosulphate titrant**:- Dissolve 6.205g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute it to 1 lit. Standardize it using standard potassium dichromate solution.
- vi) **Standard potassium dichromate** solution (0.025N):- Dissolve 1.22g $\text{K}_2\text{Cr}_2\text{O}_7$ in distilled water and dilute it to 1 lit.

PROCEDURE:-

Standardization of sodium thiosulphate titrant:-

- a) Take 100ml distilled water in a flask and add 10ml KI solution. Add 10ml 1+9 H₂SO₄.
- b) Add 20ml K₂CR₂O₇ solution and store in dark place for five minute.
- c) Titrate with Na₂S₂O₃ titrant, adding starch towards the end when a pale straw colour develops. Continue titration to the colourless end point. Note down the volume of titrant as V₂ ml.
- d) Determine the normality of the titrant 'N₂' using the relation $N_1V_1 = N_2V_2$ where
N₁ = normality K₂ CR₂ O₇ solution = 0.02
V₁ = volume of K₂ CR₂ O₇ = 20ml

Preparation of dilution water and placing it in BOD bottle

- a) Aerate the distilled water saturation with D.O. Take 1 lit of this dilution water and 1ml of each of phosphate buffer, MgSo₄, CaCl₂ and FeCl₂ solutions. Stir to mix the contents.
- b) Preparing samples of different dilution. The following dilutions are recommended depending upon the expected BOD of the waste water sample.
25%, 40%, 60%, 75% and 100%

To make dilution of say 25%, take 1 lit measuring cylinder and fill it about half with dilution water by siphoning. Add 250ml sample and make it 1 lit with dilution water stir well to mix properly. Transfer this by siphon into two BOD bottles.

- c) Repeat the procedure for other dilutions.
- d) Place one bottle in the incubator and test the second bottle immediately for D.O. using Azide – Modification method discussed later. Let this value be "D₁".
- e) Incubate the sample for 5 days at 20⁰C and test for D.O. again. Let this value be "D₂".

Alternate method of diluting the sample:-

In this method, the sample sewage is diluted to the required extent by direct pipetting a given volume of sewage into a 300ml BOD bottle and filling the remaining volume of bottle with dilution water and calculating the dilution factor. For example, if 5ml of waste water is placed in the BOD bottle and mixed with dilution water to make 300ml diluted sample, the dilution ratio will be equal to $300/5=60$. The following table gives the range of BOD that can be measured with various dilutions expressed either percentage mixtures or as ml directly pipetted into 300ml BOD bottles.

By using % mixture		By direct pipetting into 300ml BOD bottle	
% mixing	Range of BOD in mg/lit	ml	Range of BOD in mg/lit
0.01	20000-70000	0.03	30000-105000
0.02	10000-35000	0.05	12000-42000
0.05	4000-14000	0.10	6000-21000
0.10	2000-7000	0.20	3000-10500
0.20	1000-3500	0.50	1200-4200
0.50	400-1400	1.0	600-2100
1.0	200-700	2.0	300-1050
2.0	100-35	5.0	120-240
5.0	40-140	10.0	60-210
10.0	20-70	20.0	30-105
20.0	10-35	50.0	12-42
50.0	4-14	100.0	6-21
100.0	0-7	300.0	0-7

Estimation of D.O.:-

- To the sample in the BOD bottle, add 2ml manganese sulphate solution, 2ml Alkali – iodide – Azide reagent, well below the surface.
- Stopper and mix by inverting bottle at least 15 times. Let the brown precipitate settle for 5 minutes.
- Add 2ml conc. H₂SO₄ and stopper. Gently invert to dissolve the precipitate totally.
- Take 203ml of bottle water in a flask and titrate with 0.025N sodium thiosulphate solution.
- Add 1 to 2ml starch and continue titration till the first disappearance of pale blue colour.
- Note down the volume of titrant used upto colourless end point.

Note:- 200ml of sample have to be used in the titration. The volume corresponding to 200ml of original sample after correction for a total of 4ml (2ml manganese solution and 2ml alkali-azide reagent) in a 300 ml bottle is

$$\frac{200 \times 300}{(300-4)} = 202.7 \text{ml} = 203 \text{ml.}$$

OBSERVATIONS:-

S.No	Burette Reading		Vol. of Titrant used
	Initial	Final	

CALCULATIONS:

Initial D.O. of dilute sample (before incubation)
In mg/l = D_1 = ml of 0.025N sodium thiosulphate used.

Final D.O. of dilute sample (after incubation)
In mg/l = D_2 = ml of 0.025N sodium thiosulphate used.

BOD of the sample in mg/l = $\frac{D_1 - D_2}{P}$ (if dilution is expressed in percentage)

Where P = Decimal fraction of sample used.

BOD of the sample in mg/l = $(D_1 - D_2) \times$ dilution ratio (if sample is directly pipetted into BOD bottle)

Where dilution ratio = $\frac{\text{Volume of diluted sample}}{\text{Volume of undiluted sewage sample}}$

RESULTS:-

INTERPRETATION OF RESULTS:

DISSOLVED OXYGEN

AIM:- To determine the concentration of dissolved oxygen in the sample water by Azide modification method.

APPARATUS:- Glassware along with BOD bottle.

THEORY:- Dissolved oxygen is one of the most important constituents of natural water system. A certain amount of dissolved oxygen is essential for aerobic decomposition of waste to avoid nuisance conditions in rivers and to maintain the growth of fish and other aquatic animals. Oxygen is generally absorbed by water from atmosphere and unpolluted natural surface waters are usually saturated with it. Except oxygen the presence of any other gas dissolved in water is not desirable and steps should be taken to remove the same. This test is the basis of BOD test, which is an important parameter to evaluate pollution potential of the wastes. This test determines the quality of raw water and keeps proper check on stream pollution. Dissolved oxygen in the water is determined by the modified winkler's method, also called Azide modification method, whose principle is discussed below.

Dissolved oxygen present in the sample water oxidizes manganous sulphate to produce manganese hydroxide (a brown colour precipitate) after addition of NaOH and KI. Upon acidification, manganese reverts to its manganous form and liberates iodine from KI, equivalent to dissolved oxygen content in the sample water. The liberated iodine is titrated against sodium thiosulphate using starch as indicator.

RELEVANCE:- The presence of dissolved oxygen in water near its saturation level is an indication of its purity. A stream must have a minimum of 2 ppm of dissolved oxygen to support fish and other higher life forms. On the other hand, high amount of dissolved oxygen corrodes the distribution system. The permissible limit of dissolved oxygen in the domestic water supplies is 5-6 ppm.

REAGENTS:-

1. **Manganese sulphate solution**:- Dissolve 480 grams $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ in distilled water and make it to 1 lit.

2. **Alkali – Iodide – Azide reagent**:- Dissolve 500 gram NaOH and 150 gram KI in distilled water and dilute to 1 lit. Add 10 gram sodium Azide (NaN_3) dissolved in 40ml distilled.

3. **Concentrated H_2SO_4**

4. **Starch**:- Dissolve 5grams starch in 1 lit boiling distilled water. Preserve by adding a few drops of Toluene.

5. **Sodium Thiosulphate solution**: Dissolved 6.205 grams $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute it to 1 lit. Standardize it using potassium dichromate solution.

6. **Standard potassium dichromate solution**:- Dissolve 1.226 grams $\text{K}_2\text{Cr}_2\text{O}_7$ in distilled water and dilute it to 1 lit.

7. **Potassium Iodide Solution**:- Dissolve 20 grams KI in 100ml distilled water.

PROCEDURE:-

1. Standardization of sodium Thiosiphate Titrant:-

- Take 100 ml distilled water in a flask and add 10ml KI solution. Add 10ml 1+9 H₂SO₄.
- Add 20ml K₂CR₂O₇ solution and store it in a dark place for five minutes.
- Titrate with Na₂S₂O₃ titrant, adding starch towards the end when a pale straw colour develops. Continue titration to colourless end point. Note down the volume of titrant used.
- Determine the normality of titrant using the relation $N_1V_1 = N_2V_2$

N_1 = normality of K₂CR₂O₇ = 0.025

V_1 = volume of K₂CR₂O₇ – 20ml

V_2 = volume of Titrant consumed

N_2 = normality Titrant (unknown)

Adjust the normality of Titrant to 0.025N using the relation $N_1V_1 = N_2V_2$.

2. Estimation of D.O. in the sample water:-

- To the sample in the BOD bottle, add 2ml manganese sulphate solution, 2ml of alkali azide reagent, well below the surface.
- Stopper and mix by inverting bottle at least 15 times. Let the brown ppt settle for 5 minutes.
- Add 2ml conc. H₂So₄ and stopper. Gently invert to dissolve the ppt totally.
- Take 203ml of the bottle water in the flask and titrate with sodium thiosulphate titrant.
- Add 1-2 ml starch and continue titration till the first disappearance of pale blue colour.

OBSERVATIONS:-

S.No	Burette Readings		Vol. of Titrant used
	Initial	Final	

CALCULATIONS:-

D.O. in mg/l = ml of 0.025N sodium thiosulphate used.

RESULTS:-

INTERPRETATION OF RESULTS:-

Chemical Oxygen Demand (C.O.D)

Aim: To determine chemical oxygen demand (C.O.D.) of the given water sample as per IS: 3025 (Part 58)

Apparatus: C.O.D. digester, COD vials, Glassware

Chemicals:

- Potassium dichromate
- Sulfuric acid
- Ferrous Ammonium Sulphate (FAS)
- Silver Sulphate
- Mercury sulphate
- Ferroin indicator
- Organic free distilled water

Preparation of reagents:

- Standard Potassium Dichromate reagent (used as digestion solution). It is prepared by:
 - 4.913 g $K_2Cr_2O_7$ + 33g Mercuric sulphate + 167 ml conc. H_2SO_4 + make-up this mixture to 1000 ml using distilled water. This is standard potassium dichromate solution used for dilution
- Sulphuric acid reagent - catalyst solution. It is prepared by:
 - 5.5g silver sulphate crystals + 500 ml conc. H_2SO_4 for 24 hours to dissolve silver sulfate crystals completely
- Standard Ferrous Ammonium Sulphate Solution
 - Dissolve 39.2g Ferrous ammonium Sulphate crystals in 1000ml distilled water

Procedure:

- Take two COD vials with stopper (One for sample and one for blank)
- Add 2.5 ml sample and 2.5 ml D.W to each vial respectively
- To each vial, add 1.5 ml potassium dichromate solution (digestion solution)
- Add 3.5 ml sulphuric acid reagent (catalyst solution) [COD vials are HOT now]
- Cap the vials TIGHTLY and place them in the digester with the temperature set to 150C for 120 minutes. Start the digester.
- After two hours, transfer the cooled digested solution to a conical flask
- Add few drops of ferroin indicator to the flask. The solution turns bluish green
- Fill the burette with Ferrous Ammonium SULPHATE (FAS)
- Titrate the solution in the flask with FAS (in the burette)

•Appearance of reddish brown colour indicates end point of the titration. Note the volume of FAS consumed.

COD is determined using the formula:

COD in mg/L = $[(A-B) * N * 8 * 1000] / [\text{volume of sample taken}]$

(OBSERVATIONS)

Result:

Notes:

- 1.COD does not differentiate between biologically available and inert organic matter.
- 2.COD is a measure of total quantity of oxygen needed to oxidise ALL organic matter into carbondioxide and water
- 3.COD values are ALWAYS GREATER THAN BOD values
- 4.Potassium dichromate is considered the best oxidising agent
- 5.Mercuric sulphate is added to reduce the interference of chlorides
- 6.For industrial wastewater COD is almost 2.5 times BOD

MEASUREMENT OF TURBIDITY USING NEPHELOMETER

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

APPARATUS:- Nephelo turbidity meter.

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

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

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

REAGENTS:-

1. **Turbidity free water:-** Pass distilled water through a lower turbidity than distilled water, discard the first 200ml, collected. If filtration does not reduce turbidity use distilled water.

2. Stock turbidity solutions:-

i) **Solution 1:-** Dissolve 1.0 grams hydrazine sulfate $(\text{NH}_2)_2 \cdot \text{H}_2\text{SO}_4$ in distilled water and dilute it to 100 ml in a make up flask.

ii) **Solution 2:-** Dissolve 10.0 grams hexamethylene tetramine $(\text{CH}_2)_6\text{N}_4$ in distilled water and dilute it to 100ml.

iii) **Solution 3:-** In a 100ml flask, mix 5ml. each of solution 1 and 2. Allow it to stand 24 hours, then dilute it to 100ml and mix thoroughly. The turbidity of this solution is 400 NTU.

iv) **Standard Turbidity Solution:-** Take 10.0ml of solution 3 in a 100ml make up flask and dilute it to 100ml. with turbid free water. The turbidity of this suspension is 40 NTU.

PROCEDURE:-

a) Calibration of Nephelometer:-

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

b) Determination of turbidity of sample water:

- i) For samples having turbidities less than 40 NTU: Thoroughly shake the sample so as to remove any air bubbles and pour it into meter cell. Read out the turbidity of the sample from the digital display.
- ii) For samples having turbidities above 40 NTU:- Dilute sample with 1,2 or 3 volumes of turbidity free water and convert the value obtained as below.

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

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

Where

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

C = Sample volume for dilution in ml.

OBSERVATIONS:-

For undiluted sample

Digital read out =

For diluted sample

Vol. of sample (C) =

Vol. of dilution water (B) =

Digital read out (A) =

CALCULATIONS:-

For undiluted sample

Turbidity of sample in NTU =

For diluted sample

Turbidity in NTU = $\frac{A(B + C)}{C}$

RESULTS:-

INTERPRETATION OF RESULTS:-

DETERMINATION OF OPTIMUM COAGULANT DOSE USING JAR TEST APPARATUS

AIM:- To determine the optimum coagulant dose.

APPARATUS:- Jar test apparatus and Nephelo turbidity meter.

THEORY:- In plain sedimentation, very fine suspended particles of size 0.006mm to 0.002mm are not removed, since they required a detention period of 10 hours to 4 days which is impracticable. In addition to this fine suspended particle, water also contains electrically charged colloidal particles which are continuously in motion and never settle down due to gravity. It has been found that, the above mentioned impurities can be removed by sedimentation with coagulation.

It has been found that when certain chemicals (i.e. coagulants) are added to water an insoluble, gelatinous precipitate is formed. This precipitate during its formation and descent through the water absorbs very fine suspended and colloidal impurities there by reducing the turbidity of the water.

RELEVANCE:-

Coagulation of raw water using the optimum coagulant dose removes colloidal impurities from the water. These colloidal impurities are normally associated with organic matter containing pathogenic bacteria which are responsible for water borne diseases. The chemical coagulation also makes the process of disinfection more effective. Coagulation also removes objectionable colour, taste and odour's from water. Usually the dose of Alum varies between 5mg/lit for relatively clear water to about 85 mg/lit for very turbid waters. The average dose is about 20mg/lit.

CHEMICAL REAGENTS:-

ALUM SOLUTIONS:- Dissolve 1.0 gram of Alum in 1 lit of distilled water so that each ml. of Alum solution contains one milligram of Alum.

PROCEDURE:- Take 2 lit of sample water in all the six jars of the apparatus. Then add Alum solution in each of the six jars in varying amounts. The range of Alum dose depends upon the turbidity of the raw sample water. The normal range of Alum dose varies between 15ppm to 60ppm. Add the Alum solution to each of the six jars as per the tabular column shown in observation sheet. After adding different amounts of Alum solution in the all six jars, place the jars on the platform provided and fix the stirring paddle to the connecting rod which rotates by a gear and spindle system, with thee help of electric motor, the paddles are rotated at a speed of 30-40 rpm for about 2 minutes. The speed of the paddles are then reduced to a minimum so as to cause flocculation and stirring is continued to about 20-30 min. the rotation of paddles are then stopped and floc is allowed to settle for 30min. then pippet out the supematant from each jar and measure the turbidity using nephelometer.

GRAPH:- Plot a graph between the coagulant dose applied and turbidity of coagulated sample, by taking turbidity value on Y-axis and Alum dose on X-axis; as shown below. Then determine optimum coagulant dose from the graph which corresponds to minimum turbidity.

OBSERVATIONS:

Jar No.	Alum dose in mg/lit	ml of Alum solution to be added in 2000ml

CALCULATIONS:-

RESULTS:-

INTERPRETATION OF RESULTS:-

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CALCULATIONS:-

RESULTS:-

INTERPRETATION OF RESULTS:-

RESIDUAL CHLORINE

AIM:- To determine the amount of residual chlorine in the chlorinated water sample.

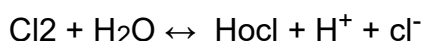
APPARATUS:-

(i) Glass ware (ii) Coloroscope

THEORY:-

Chlorination is a common and widely used method of disinfection of water. Water is normally chlorinated to an extent beyond a little beyond the break point. This ensures the availability of free residual in the treated water. The dosage of chlorine should be such that residual chlorine of 0.1 to 0.2 ppm appears in water at the point of its entry into the distribution system. Free residual chlorine (i.e. HOCl and OCl⁻) is more effective than combined residual (i.e. chloramines). Hence water should be treated beyond breaking point to ensure the presence of free residual chlorine.

When chlorine is added to water, it reacts with water forming Hypochlorous acid. This is indicated by the following reaction.



The above reaction is reversible. The hypochlorous acid (HOCl) dissociates into hydrogen ions (H⁺) and hypochlorite ions (OCl⁻) as indicated below.



It is the hypochlorous acid (HOCl) and the hypochlorite ions, which accomplish disinfection of water. The hypochlorous acid (HOCl) is about 80 times more powerful as a disinfectant than the hypochlorite ions (OCl⁻). The dissociation of hypochlorous acid is a function of the pH value of chlorinated water. As the pH value of water increases, more and more HOCl dissociates to form OCl⁻ ions. At a pH of about 5.5, hypochlorous acid is 100% unionized while at pH above 9.5 it is all OCl⁻ ions. Therefore chlorination should be carried out at low pH so as to ensure more percentage of hypochlorous acid for effective chlorination.

Residual chlorine is estimated by two methods.

(i) Starch – Iodide method (ii) O – Tolidine method.

In starch-iodide method, chlorine displaces an equivalent amount of iodine from iodide (i.e. KI) which combines with starch to give blue colour. The liberated iodine is titrated against standard sodium thiosulphate until the blue colour disappears.

O – Tolidine method is most commonly used to determine the residual chlorine in small concentrations (i.e. up to 2 ppm). O-Tolidine is an organic compound, which is oxidized by chlorine in a yellow coloured compound called "Holoquinone". Thus when O – Tolidine is added to water containing chlorine a yellow colour develops, the intensity of

which is proportional to the amount of residual chlorine present in water. The colour so produced is matched or compared with standards.

RELEVANCE:- Presence of residual chlorine (i.e. free residual) in the desired concentration ensures the safety or purity of water during its conveyance. It safeguards the water against any contamination which is likely to occur during conveyance through a network of distribution system. The permissible limit of residual chlorine in the treated water is upto 0.2 ppm.

REAGENTS:-

- i) **Potassium Iodide Solution:** Dissolve 20.0 grams KI in 100ml distilled water.
- ii) **Sodium thiosulphate titrant:** Dissolve 6.205 gram sodium thiosulphate in distilled water and make it to 1 lit. Standardize it using potassium dichromate.
- iii) **Potassium dichromate solution of 0.1N:** Dissolve 1.226 gram $K_2CR_2O_7$ in distilled water and make it to 1 lit.
- iv) **Starch:** Dissolve 5 grams starch in 1 lit boiling distilled water.
- v) **Conc.:** Acetic Acid.
- vi) **O – Tolidine reagent:** (for O – Tolidine method)

PROCEDURE:- 1. Starch – Iodide method.

Standardization of sodium thiosulphate using standard 0.1N potassium dichromate solution

- a) To 80ml distilled water, add 1ml conc. H_2SO_4 , 10ml 0.1N $K_2CR_2O_7$ and 10ml KI solution.
- b) Keep the flaks in the dark place for about 5 min.
- c) Titrate with the given sodium thiosulphate till a pale yellow colour is observed. At this juncture, add 1ml starch and continue titration until blue colour disappears. Note down the volume of sodium thiosulphate consumed.

$$\text{Normality of sodium thiosulphate} = \frac{1}{\text{ml of sodium thiosulphate consumed}}$$

Adjust the normality of sodium thiosulphate to 0.0.25 using the relation

$$N_1V_1 = N_2V_2$$

Determination of residual chlorine in the sample water:-

- a) To 100ml chlorinated water samples add 5ml. concentrated acetic acid and 10ml KI.
- b) Titrate with 0.025N sodium thiosulphate till a pale yellow colour is reached. At this juncture, add 1ml starch and continue titration till blue colour is discharged.
- c) Note down the volume of 0.025N sodium thiosulphate consumed.

Determination of Residual chlorine (O – Tolidine method)

Take 10ml chlorinated water sample in a glass tube and add to this 0.1ml O-Tolidine solution. The sample turns to yellow. Determine the concentration of residual chlorine in the sample water by comparing the colour produced with the standards (i.e. coloured glass standards tinted for each concentration).

OBSERVATION:- Starch Iodide Method

S.No	Burette Readings		Vol. of Titrant used
	Initial	Final	

CALCULATIONS:-

Starch-iodide method:-

Normality of given sodium thiosulphate titrant = $\frac{1}{\text{MI of sodium thiosulphate consumed}}$

Adjusting the normality of sodium thiosulphate to 0.025, using the relation

$$N_1V_1 = N_2V_2$$

Residual chlorine in mg/l = $\frac{\text{vol. of sodium thiosulphate consumed} * N * 35450}{\text{ml of sample water}}$

RESULT:-

By Starch – Iodide Method

Residual chlorine in mg/l = _____

By O – Tolidine method

Residual chlorine in mg/l = _____

INTERPRETATION OF RESULTS:-