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**ENVIROMENTAL
ENGG.LAB MANUAL**

LIST OF EXPERIMENTS

- Determination of Turbidity of water by using suitable method
- Determination of pH of given water sample.
- Determination of Hardness of given water sample.
- Determination of Residual Chlorine in given sample of water
- Determination of Total Suspended and Dissolved Solids in given water sample.
- Determination of Bio –chemical oxygen demand of waste water sample.
- Determination of chemical oxygen demand of waste water sample.
- Determination of Conductivity of given water sample.
- Determination of Chlorides of given water sample
- Determination of Alkalinity and Acidity of a given water sample.
- Determination of Dissolved Oxygen of given waste water sample.

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EXPERIMENT NO - 1

Aim: - Determination of Turbidity of water by using suitable method

Determining the turbidity of water is an important process that helps to ensure the safety and quality of drinking water. Turbidity is a measure of the cloudiness of water caused by suspended particles, such as silt, clay, and microscopic organisms. In this manual, we will discuss the steps involved in determining the turbidity of water using a turbidimeter.

Materials:

- Turbidimeter
- Water sample
- Calibration standard (optional)

Procedure:

- Turn on the turbidimeter and allow it to warm up for a few minutes.
- Fill a clean and clear cuvette (a small tube used for holding samples) with the water sample to be tested. If the water is cloudy, let it settle for a few minutes to allow the particles to settle to the bottom of the container.
- Wipe the cuvette clean with a lint-free cloth to remove any fingerprints or smudges.
- Insert the cuvette into the turbidimeter and make sure it is positioned correctly.
- Follow the instructions for the particular turbidimeter you are using to obtain a reading. Generally, this involves pressing a button or turning a dial to initiate the reading.
- Record the turbidity value displayed on the turbidimeter.
- If desired, calibrate the turbidimeter using a calibration standard to ensure accurate readings. Follow the manufacturer's instructions for calibration.

Tips:

- Make sure to use a clean and clear cuvette for each water sample to avoid contamination and inaccurate readings.
- If the water sample is too cloudy to get an accurate reading, dilute it with distilled water before testing.
- Turbidity values are usually reported in nephelometric turbidity units (NTU). The higher the NTU value, the cloudier the water.

Safety Precautions:

- Always handle water samples with care and follow appropriate safety procedures.
- If you are working with potentially hazardous water samples, wear appropriate personal protective equipment (PPE), such as gloves and safety glasses.

In conclusion, determining the turbidity of water is a simple but important process for ensuring the safety and quality of drinking water. By following the steps outlined in this manual, you can obtain accurate turbidity readings and help ensure the safety of the water supply.



EXPERIMENT NO – 2

Aim: - Determination of pH of given water sample

Theory : - Determining the pH of a water sample is a straightforward process that involves the use of pH indicator strips or a pH meter. Below is a step-by-step guide on how to determine the pH of a given water sample:

Apparatus: -

- Water sample
- pH indicator strips or pH meter
- Beaker or test tube
- Stirring rod (if using pH indicator strips)
- pH chart (if using pH indicator strip)



Steps:

- Collect the water sample you want to test in a clean beaker or test tube.

- If using pH indicator strips, dip the strip into the water sample and hold it in the liquid for a few seconds. If using a pH meter, dip the probe into the water sample and wait for a reading to stabilize.
- If using pH indicator strips, remove the strip from the water sample and shake off any excess liquid. Then, compare the color of the strip to the pH chart to determine the pH of the water sample. Note that different pH indicator strips may have different color scales, so be sure to use the chart that came with your strips.
- If using a pH meter, read the pH value on the display. Some pH meters may require calibration before use, so be sure to follow the manufacturer's instructions.
- Record the pH value of the water sample.
- If using pH indicator strips, rinse the strip with distilled water and air dry it before storing. If using a pH meter, clean the probe with distilled water and store it according to the manufacturer's instructions.

It's important to note that the pH of water can be affected by various factors, such as dissolved solids, temperature, and the presence of contaminants. Therefore, it's best to test water samples under consistent conditions, such as at room temperature and after filtration. Additionally, pH readings should be interpreted in the context of the source and intended use of the water sample. For example, drinking water should have a pH of 6.5 to 8.5 for optimal health and taste.

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EXPERIMENT NO – 3

Aim: - Determination of Hardness of given water sample.

THEORY:

Hardness is deemed to be the capacity of water for reducing and destroying the lather of soap. Hardness in water is due to the natural accumulation of salts from contact with soil geological formations or it may enter from direct pollution by industrial effluents. Calcium and magnesium are the principle cations causing hardness. Iron, aluminum, manganese, strontium and zinc also cause hardness but to a relatively little extent or to negligible amount. The term “Total Hardness” indicates the concentration of calcium and magnesium ions only. However, if present in significant amounts the other metallic ions should also be included. The total hardness is expressed in terms of calcium carbonate.

Calcium and magnesium ions react with EDTA to form soluble complexes and the completion of reaction is indicated by the colour change of a suitable indicator such as Eriochrome black T.

APPARATUS:

- Burette with stand Pipette
- Two Conical Flasks
- Measuring Cylinder

REAGENTS:

- **Standard N/50 (0.02N) EDTA Solution:** Dissolve 40gm of sodium salt of EDTA and 0.1gm $MgCl_2$ in 800ml of distilled water. Standardize with $CaCl_2$ solution.
- **Eriochrome Black T indicator:** Dissolve 0.1gm of Eriochrome black T in 20ml of ethyl alcohol.

- **Ammonia Buffer Solution:** Dissolve 6.75gm of ammonium chloride in 75ml of liquid ammonia and dilute to 100ml distilled water.

PROCEDURE:

- Take 25ml of water sample in a conical flask.
- Add 1ml of Ammonia buffer to the flask,
- Add a pinch of Eriochrome Black T indicator to the flask. Wine red colour will be developed.
- Titrate with standard EDTA solution (to be filled in burette) till the colour changes from wine red to blue.

CALCULATIONS:

Normality of EDTA = 0.02N

TOTAL HARDNESS

SAMPLE	INITIAL READING	FINAL READING	VOLUME USED OF EDTA

PERMANENT HARDNESS

SAMPLE	INITIAL READING	FINAL READING	VOLUME USED OF EDTA

Total Hardness (as CaCO_3)

$\text{mg/l} = (\text{Volume of EDTA titrant} \times N \times 1000) / (\text{Volume of Sample taken for Titration})$

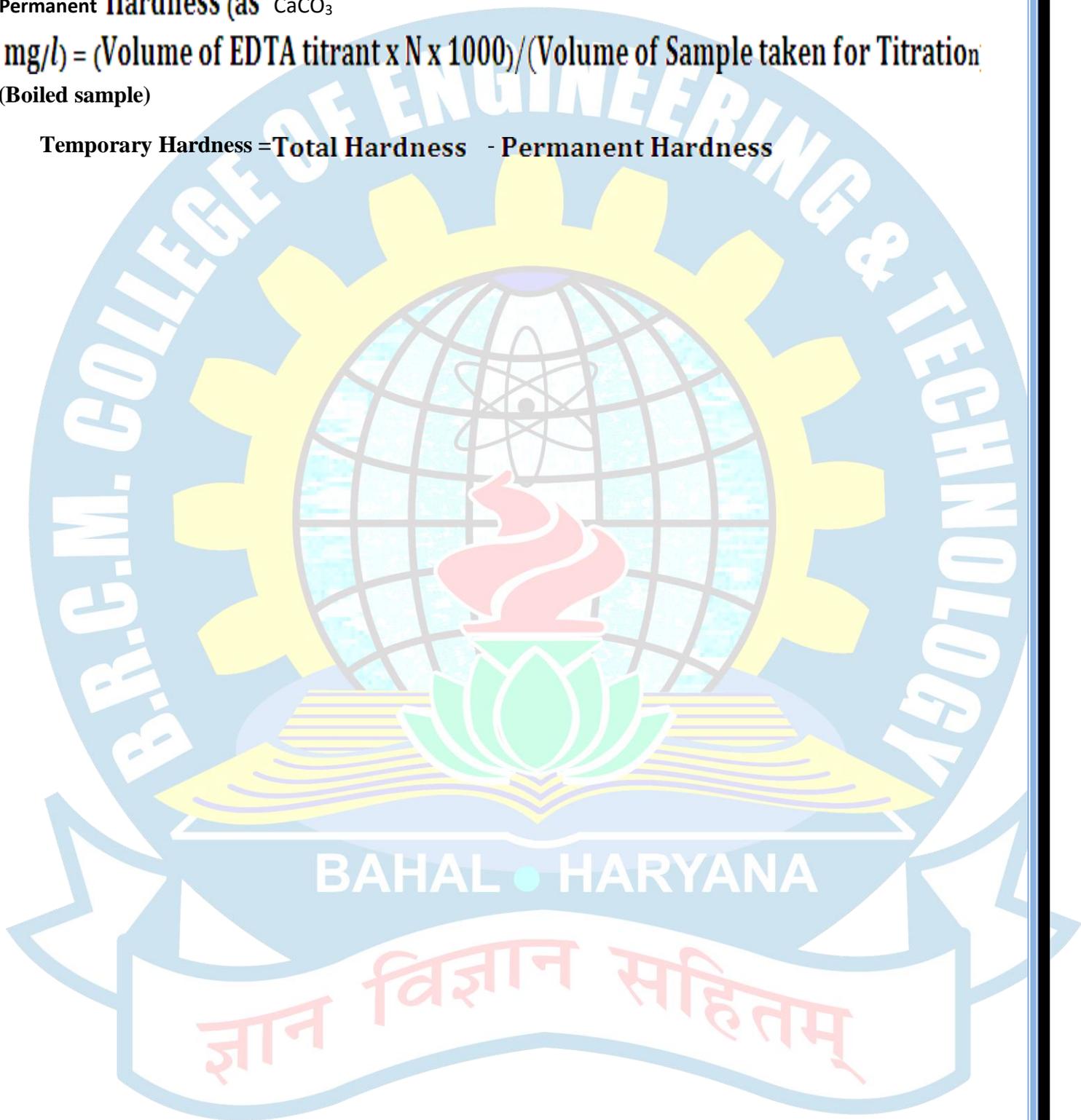
(Unboiled sample)

Permanent Hardness (as CaCO_3

$$\text{mg/l) = (Volume of EDTA titrant} \times N \times 1000) / (\text{Volume of Sample taken for Titration})$$

(Boiled sample)

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



EXPERIMENT NO - 4

AIM: Determination of Residual Chlorine in given sample of water

Introduction: Residual chlorine is the amount of chlorine remaining in water after the disinfection process. It is an important parameter to determine the effectiveness of the disinfection process and to ensure that the water is safe for consumption. The purpose of this lab is to determine the residual chlorine in a given sample of water using the standard method of titration with a sodium thiosulfate solution.

Materials:

- Water sample containing residual chlorine
- Potassium iodide solution (10% w/v)
- Sodium thiosulfate solution (0.1 N)
- Starch solution (1% w/v)
- 250 mL Erlenmeyer flask
- 50 mL burette
- Pipette (10 mL)
- Graduated cylinder (50 mL)
- Glass rod
- Burette clamp
- White tile
- Funnel

Procedure:

- Rinse the burette with distilled water and then with the sodium thiosulfate solution.
- Fill the burette with the sodium thiosulfate solution up to the zero mark.

- Pipette 10 mL of the water sample into the Erlenmeyer flask.
- Add 10 mL of the potassium iodide solution to the Erlenmeyer flask.
- Add a few drops of the starch solution to the Erlenmeyer flask.
- Swirl the Erlenmeyer flask to mix the contents.
- Titrate the mixture in the Erlenmeyer flask with the sodium thiosulfate solution from the burette until the blue color of the iodine-starch complex disappears and turns into a colorless solution.
- Record the volume of the sodium thiosulfate solution used for titration.
- Repeat steps 3-8 for two more trials.
- Calculate the average volume of the sodium thiosulfate solution used for titration.
- Calculate the residual chlorine concentration in the water sample using the following formula:

Residual chlorine concentration (mg/L) = [(Volume of sodium thiosulfate solution used x Normality of sodium thiosulfate solution x 71)/(Volume of water sample taken x 1000)] x 1000

Where 71 is the molecular weight of chlorine.

Results: Record the volume of the sodium thiosulfate solution used for titration for each trial in a table. Calculate the average volume and the residual chlorine concentration for the water sample.

Discussion: Discuss the accuracy and precision of the results obtained. Compare the residual chlorine concentration in the water sample with the acceptable limits set by regulatory authorities. Interpret the significance of the results in terms of the safety and suitability of the water sample for consumption.

Conclusion: In conclusion, the residual chlorine in the given water sample was determined using the standard method of titration with sodium thiosulfate solution. The results showed that the residual chlorine concentration was within acceptable limits set by regulatory authorities. Therefore, the water sample is safe and suitable for consumption.

EXPERIMENT NO - 5

AIM: Determination of Total Suspended and Dissolved Solids in given water sample.

Theory:-

The total amount of solids (TS) includes the suspended solids (SS) as well as the dissolved solids. The total solids in a waste water sample can be determined by evaporating the sample and weighing the dry residue left. The suspended solids can be found by filtering the sample weighing the dry residue left on the filter paper. The difference between total solids and suspended solids will represent nothing but the dissolved solids.

The dissolved solids, which usually predominate in waters, consist mainly of inorganic salts, small amount of organic matter and dissolved gases. The suspended solids contain much of the organic matter and any increase thereof tends to increase the degree of pollution in water.

The amount of total solids (TS) in given water up to 500mg/l generally makes it suitable for domestic uses. Waters with higher T.S. contents up to about 1000mg/l, may also be acceptable, although may cause some psychological effects on some human beings, consuming such a water. High concentrations of dissolved solids in waters when used in boilers may lead to boiler troubles lead priming and foaming.

APPARATUS:

- Beaker
- Measuring Cylinder
- Heating Plate
- Weighing Machine with weights
- Filter paper
- Funnel Oven Petri plate

PROCEDURE:

- Weigh the Petriplate (clean and dry) and record its mass as A gm.
- Fill 50ml of water sample in the petriplate.
- Place the petriplate on heating plate and evaporate the sample to dryness.
- After cooling the beaker, record the mass of the petriplate with dry residue in gm. Let it be B gm.
- Take another 50ml of sample and given the standard filter paper.
- Record the mass of the filter paper. Let it be C gm.
- Filter the sample through the filter paper in a funnel.
- Place the filter paper with residue in an oven and evaporate it to the dryness.
- Record the mass of the filter paper and residue as D gm.

CALCULATION:

Total Solids

Sample Volume = 50 ml

Mass of empty petriplate (A) = gm

Mass of empty petriplate + Residue = gm

(Y) Concentration = (B-A) = 44.63-44.61 = gm

$$\text{Total Solids (mg/l)} = \frac{(B - A) \times 1000}{\text{Volume of Sample taken}}$$

Volume of Sample taken

Suspended Solids

Mass of filter paper (C) = gm

Mass of filter paper + Residue (D) = gm

(X) Concentration = (D-C) = 0.90-0.89 = gm

$$\text{Total Solids (mg/l)} = \frac{(D - C) \times 1000}{\text{Volume of Sample taken}}$$

Total Dissolved Solids = (Y-X) = Total Solid-Suspended Solid

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EXPERIMENT NO - 6

Aim: - Determination of Bio –chemical oxygen demand of waste water sample.

THEORY:

The biochemical oxygen demand (BOD) is the amount of oxygen required by bacteria while stabilizing decomposable organic matter under aerobic conditions. The quantity of oxygen required may be taken as a measure of its content of decomposable organic matter. The rate of BOD exertion is governed by the characteristics of sewage. Its decomposable organic content, bacterial population and temperature. The progressive BOD exertion takes place in two stages.

(a) Carbonaceous, (b) Nitrification

It has been observed the large percentage of total BOD is exerted in 5 days at 20°C. The value of 5 days at 20°C is to a reasonable extent comparable to 4 days at 30°C and 3 days at 35°C.

$$Y = L(1 - 10^{-kt})$$

$$Y = \text{BOD at any time} \quad L = \text{Ultimate BOD}$$

$$k = \text{reaction rate} \quad t = \text{time}$$

REAGENTS:

(i) Phosphate buffer	(v) Manganous sulphate solution
(ii) Magnesium sulphate solution	(vi) Alkaline Potassium iodide solution
(iii) Calcium chloride solution	(vii) N/40 sodium thiosulphate solution
(iv) Ferric chloride solution	(viii) Conc. sulphuric acid
	(ix) Starch indicator

BOD MEASURABLE WITH VARIOUS DILUTIONS

Range of BOD	% mixture
200-700	1.00
100-350	2.00
40-140	5.00
20-70	10.00
10-35	20.00
4-14	50.00

PROCEDURE:

Prepare dilution water by adding 1.0ml each of phosphate buffer solution, magnesium sulphate solution, calcium chloride and ferric chloride solution to 1.0litre of distilled water. Add 2.0mlsettled sewage and aerate. Determine the exact capacity of three BOD capacities of three BOD bottles. Find out the D.O. of undiluted sample as in 4.8 and designate as DO.

Prepare the desired percent mixture by adding sample in dilution water. Fill up one bottle with the mixture and the other one with dilution water (blank). Incubate at a fixed temperature for a definite time (20°C, 5 days). Find out DO in both the bottles after incubation and designate.

Mixture as (DO₁)
Blank (DO_b)

CALCULATIONS:

$$\text{BOD mg/l} = [(DO_b - DO_1) 100\%] - (DO_b - DO_s)$$

OBSERVATIONS:

Sample details Source	% mixture	DO _s	DO _b	DO ₁	BOD mg/l

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EXPERIMENT NO – 7

Object:- Determination of chemical oxygen demand of waste water sample.

Theory:-

Chemical oxygen demand test is widely used for measuring the pollutional strength of waste waters. All organic compounds with a few exceptions can be oxidized to carbon dioxide and water by the action of strong oxidizing agents regardless of biological assimilability of the substances.

REAGENTS:

- Standard potassium dichromate 0.25 N
- Sulphuric acid (with 1gm of silver sulphate in every 75ml acid)
- Ferrion indicator solution
- Standard ferrous ammonium sulphate solution

STANDARDIZATION PROCEDURE:

- Dilute 25ml standard potassium dichromate solution to about 250ml with distilled water. Add 20ml cone. Sulphuric acid, Titrate with ferrous ammonium sulphate solution using ferroin indicator to red end point.

$$\text{Vol. of } K_2Cr_2O_7 \times 0.25$$

- Normality of $FeSO_4(NH_4)_2SO_4 = \frac{\text{Volume of } FeSO_4(NH_4)_2SO_4 \text{ Used}}{\text{Vol. of } K_2Cr_2O_7 \times 0.25}$

PROCEDURE:

- Place 50ml or fraction diluted to 100ml of sample with distilled water in hard glass bottle and add 25ml standard potassium dichromate solution. Carefully add 75ml cone. H_2SO_4 mixing after each addition. Digest the mixture in pressure cooker or autoclave for 30 min.
- Repeat the procedure with 100ml distilled water and reagents as in (a).

- Transfer the contents to a 500ml conical flask. Dilute the mixture to about 350ml. Titrate the excess dichromate with standard ferrous ammonium sulphate using ferroin indicator. The end point is red. Designate the titration value for sample (a) as B and for distilled (b) as A.

CALCULATIONS:

Where

$COD\ mg/l = (A-B) C \times 8 \times 1000/ml\ sample$
 A = ml $FeSO_4(NH_4)_2SO_4$ used for bank
 B = ml $FeSO_4(NH_4)_2SO_4$ used for sample
 C = ml $FeSO_4(NH_4)_2SO_4$ solution determined above.

OBSERVATIONS:

Sample details		Normality of $K_2Cr_2O_7$	Amount of $K_2Cr_2O_7$ Added	Normality of $FeSO_4(NH_4)_2SO_4$	ml of $FeSO_4(NH_4)_2SO_4$ Used	COD mg/l
Source	Volume					

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EXPERIMENT NO – 8

Object:- Determination of Conductivity of given water sample.

Theory:-

Conductivity is a measure of the ability of a substance to conduct an electric current. It is an important parameter to determine the purity of water and the concentration of dissolved ions. The purpose of this lab is to determine the conductivity of a given water sample using a conductivity meter.

Apparatus:-

- Water sample
- Conductivity meter
- Calibration solution (1000 $\mu\text{S}/\text{cm}$)
- Beaker (100 mL)
- Stirrer
- Pipette (10 mL)
- Funnel

Procedure:-

- Rinse the conductivity meter with distilled water and then with the calibration solution.
- Calibrate the conductivity meter with the calibration solution according to the manufacturer's instructions.
- Pour the water sample into a clean beaker.
- Place the conductivity meter into the water sample and wait until the reading stabilizes.
- Record the conductivity reading.
- Stir the water sample gently with a stirrer to ensure uniformity of the sample.
- Repeat steps 4-6 two more times.
- Calculate the average conductivity reading of the water sample.

Results:

Record the conductivity reading for each trial in a table. Calculate the average conductivity

Discussion:

Discuss the accuracy and precision of the results obtained. Compare the conductivity reading of the water sample with the acceptable limits set by regulatory authorities. Interpret the significance of the results in terms of the purity and suitability of the water sample for consumption or industrial use.

Conclusion:

In conclusion, the conductivity of the given water sample was determined using a conductivity meter. The results showed that the conductivity reading was within acceptable limits set by regulatory authorities. Therefore, the water sample is pure and suitable for consumption or industrial use.



EXPERIMENT NO – 9

Object:- Determination of Chlorides of given water sample

Theory:-

SILVER NITRATE METHOD:

Chloride is the common anion found in water and sewage. The concentration of chloride in natural waters varies from a few milligrams to several thousand milligrams per litre. Higher concentrations of chloride may be due to the contamination by sea water, brines, sewages or industrial effluents such as those from paper works, galvanizing plants, water softening plants and petroleum refineries.

Silver nitrate reacts with chloride ions to form silver chloride. The completion of reaction is indicated by the red colour produced by the reaction of silver nitrate with potassium chromate solution which is added as an indicator.



Apparatus:-

- Burette with stand Pipette
- Measuring Cylinder (100ml)
- Two Conical Flasks
- N/35.5 AgNO₃ Solution (Silver Nitrate Solution)

REAGENTS:

Use chloride –free distilled water for the preparation of all reagents :

(i) **Standard Silver Nitrate Titrant, N/35.5 (0.0282N):** Dissolve 4.791gm silver nitrate, AgNO₃ in distilled water and make up to 1000ml in a volumetric flask. Standardize it against

0.0282N sodium chloride solutions

1.0ml of exactly 0.0282 N AgNO₃ = 1.0mg Cl

(ii) **Potassium Chromate Indicator Solution:** Dissolve 25gm potassium chromate (K₂CrO₄) in distilled water. Add silver nitrate solution drop wise until a slight red precipitate is formed. Allow to stand for 12 hours. Filter and dilute the filtrate to 500ml.

Procedure:-

- Take 25ml of sample in a conical flask.
- Place the same quantity of chloride free distilled water in another flask, to serve as a blank.
- Add to both the flasks. 10 drops of potassium chromate indicator each.
- Titrate the sample as well the distilled water (Blank) in both the flasks with N/35.5 Silver Nitrate Solution.
- Note the amount of titrant used till yellow colour in the flask is turned into reddish colour.

CALCULATION:

Equivalent Weight of Chloride = 35.45

Normality of AgNO₃ = N/35.45

$$\text{Chloride Conc. (as CaCO}_3 \text{ mg/l)} = \frac{(\text{Vol. Of AgNO}_3 - \text{Blank used}) \times \text{Normality of AgNO}_3 \times 35.45 \times 1000}{\text{Volume of Sample taken for estimation}}$$

Sample	INITIAL READING	FINAL READING	VOLUME USED OF AgNO ₃
Blank (Dist. Water)			

Sample	INITIAL READING	FINAL READING	VOLUME USED OF AgNO ₃
Sewage Sample			

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EXPERIMENT NO – 10

Object:- Determination of Alkalinity and Acidity of a given water sample.

Theory:-

PART 1:- To determine the Acidity of the given water sample.

THEORY:

Acidity is usually caused by the presence of free carbon dioxide, mineral acids such as sulphuric and weakly dissociated acids. Iron and aluminium salts hydrolyze in water to release mineral acidity. Surface waters and ground waters attain acidity from humic acids from industrial wastes such as pickling liquors and from acid mine drainage. Generally the pH of most of the samples is more than 7 and consequently carbon dioxide acidity is the only acidity present in them. Samples contaminated with acidic wastes only will have a pH below 4.5 and contain both mineral acidity and carbon dioxide acidity.

REAGENTS:

- (i) **Methyl orange indicator:** Dissolve 50mg methyl orange powder in distilled water and dilute to 100ml.
- (ii) **Phenolphthalein indicator solution:** Dissolve 500mg phenolphthalein in 50ml ethyl or isopropyl alcohol and add 50ml distilled water. Add 0.02 N sodium hydroxide solution drop wise until a faint pink colour appears.
- (iii) **Sodium hydroxide solution, 0.02 N:** Prepare sodium hydroxide solution by dissolving 40g NaOH in distilled water and diluting to 1000ml. Standardize it against standard N- sulphuric acid. Dilute appropriate volume of N-NaOH solution (about 20ml) to 1000ml to give a 0.02 N solution. 1.0ml of exactly 0.02 N-NaOH solution = 1.0mg CaCO_3 .

PROCEDURE:

- (i) **Methyl Orange Acidity:** Place 25ml of the sample in a 250ml of conical flask. Add 2 drops of methyl orange indicator solution and titrate with 0.02 N NaOH solution (to pH 4.5) to faint orange colour.
- (ii) **Total Acidity (using phenolphthalein) at room temperature:** To a suitable aliquot of the sample in a 250ml of conical flask add 3 drops phenolphthalein indicator. Titrate with 0.02 N NaOH solutions to the appearance of faint pink colour (to pH 8.3).
- (iii) **Total Acidity (using phenolphthalein) at boiling temperature:** Add 3 drops of phenolphthalein indicator solution to 25ml of sample taken in a conical flask and heat to

boiling for about 2 minutes. Titrate while hot with 0.02 N-NaOH solutions to the permanent faint pink colour.

CALCULATIONS: Normality of sodium hydroxide solution is exactly 0.02N

$$\text{Acidity as CaCO}_3 \frac{\text{mg}}{\text{l}} = \frac{\text{Volume (ml) of titrant (NaOH)} \times N \times 1000}{\text{Volume of Sample taken for Titration}}$$

AT ROOM TEMPATURE

SAMPLE	INITIAL READING	FINAL READING	VOLUME USED OF NaOH

AT BOILING TEMPERATURE

SAMPLE	INITIAL READING	FINAL READING	VOLUME USED OF NaOH

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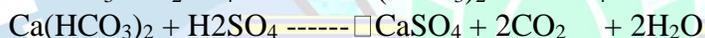
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PART 2:-To determine the Alkalinity of the given water sample.

THEORY:

Alkalinity is the quantitative capacity aqueous media to react with hydrogen ions. The alkalinity of natural and treated waters is normally due to the presence of bicarbonate, carbonate and hydroxide compounds of calcium, magnesium, sodium and potassium. Borates and phosphates and silicates also contribute to alkalinity. Some other ions not ordinarily found in natural water such as arsenate, aluminate and certain organic anions in coloured waters could also increase the alkalinity. Because of the relative abundance carbonate minerals and because of the ready availability of carbon dioxide that enters into equilibria with them in water solution, most waters contain bicarbonate and carbonates only. It is customary to calculate the hydroxide, carbonate and bicarbonate alkalinities from the titre values with a standard acid. Each form of the alkalinity exists separately or in combination. If there is phenolphthalein alkalinity it is due to hydroxide or carbonate or both. If there is methyl orange alkalinity it is due to any one of the alkalinities or hydroxides and carbonates together, or carbonate and bicarbonate together. It is to be stated that hydroxide and bicarbonate alkalinities cannot co-exist.

Alkalinity is directly determined by titration with 0.02N H₂SO₄ using phenolphthalein and methyl orange indicators.



REAGENTS:

(i) **Sulphuric acid 1N:** Place 28ml conc. H₂SO₄ in a 1000ml volumetric flask and make up to the mark with carbon dioxide free distilled water. Standardize it against 1N sodium carbonate solution using methyl orange as the indicator. (End point is the appearance of faint orange colour).

(ii) **Sulphuric acid 0.02N:** Dilute appropriate volume of 1N H₂SO₄ (about 20ml) to 1000ml in a volumetric flask with carbon di-oxide-free distilled water, to give a 0.02 N solution.



(iii) **Phenolphthalein indicator solution:** Dissolve 500mg phenolphthalein in 50ml ethyl or isopropyl alcohol and add 50ml distilled water. Add 0.02 N sodium hydroxide solution drop wise until a faint pink colour appears.

PROCEDURE:

(i) **Phenolphthalein Alkalinity (P):** Take 25ml of the sample in a 250ml of conical flask. Adjust the volume to 50ml with distilled water. Add 4 drops of phenolphthalein indicator solution. If no pink colour appears then titrate with 0.02N sulphuric acid (to pH 4.6) to

light pink.

(ii) **Methyl Orange Alkalinity (M):** Add 3 drops of methyl orange indicator in a same solution. Orange colour appears then titrates with 0.02N sulphuric acid (to pH 4.6) orange to pinkish colour.

CALCULATIONS:

PHENOLPHTHALEIN ALKALINITY (P)

SAMPLE	INITIAL READING	FINAL READING	VOLUME OF H ₂ SO ₄ USED

METHYL ORANGE ALKALINITY (M)

SAMPLE	INITIAL READING	FINAL READING	VOLUME OF H ₂ SO ₄ USED

If the sulphuric acid used for titration is exactly 0.02 N, Phenolphthalein alkalinity (as CaCO₃) mg/l

(P) Phenolphthalein Alkalinity as (CaCO₃) mg/l =

$$\frac{\text{Volume of H}_2\text{SO}_4 \text{ for Phenolphthalein end point} \times N \times 1000}{\text{Volume of sample taken for titration}}$$

(T) Total Alkalinity as (CaCO₃) mg/l =

$$\frac{\text{Volume of H}_2\text{SO}_4 \text{ for Phenolphthalein} + \text{Volume of H}_2\text{SO}_4 \text{ for Methyl orange} \times N \times 1000}{\text{Volume of sample taken for titration}}$$

Calculation of three forms of alkalinity: The titre values obtained from phenolphthalein and total alkalinity determinations are used to estimate the three forms of alkalinity as shown in the following table:

	Hydroxide-alkalinity as Ca CO ₃	Carbonate-alkalinity as CaCO ₃	Bicarbonate-alkalinity as CaCO ₃
P = 0	0	0	T
P < ½ T	0	2P	T-2P
P = ½ T	0	2P	0
P > ½ T	2P-T	2(P-T)(T-P)	0
P = T	T	0	0

Where P is phenolphthalein alkalinity and T is total alkalinity.

(It is assumed that the entire alkalinity is due to the presence of bicarbonate, carbonate and hydroxide only and other ions contributing to alkalinity are absent).

EXPERIMENT NO – 11

Object:- Determination of Dissolved Oxygen of given waste water sample.

Introduction:

Dissolved oxygen (DO) is a measure of the amount of oxygen dissolved in water. It is an important parameter to determine the quality of water and the level of pollution. The purpose of this lab is to determine the dissolved oxygen of a given waste water sample using the Winkler method.

Apparatus:-

- Waste water sample
- Sodium thiosulfate solution (0.025 N)
- Manganese sulfate solution (8.4% w/v)
- Alkaline iodide-azide reagent
- Sulfuric acid (concentrated)
- Starch solution (1% w/v)
- Burette (50 mL)
- Pipette (10 mL)
- Conical flask (300 mL)
- Funnel

Procedure:

- Rinse the conical flask with distilled water and then with the waste water sample.
- Pipette 10 mL of the waste water sample into the conical flask.
- Add 1 mL of the manganese sulfate solution to the conical flask and swirl to mix.
- Add 1 mL of the alkaline iodide-azide reagent to the conical flask and swirl to mix.
- Add 1 mL of concentrated sulfuric acid to the conical flask and swirl to mix.
- Allow the mixture to stand for 10 minutes.
- Titrate the mixture with the sodium thiosulfate solution from the burette until the yellow color of the iodine-azide complex disappears and turns into a colorless solution.
- Add a few drops of the starch solution to the conical flask.

- Continue the titration with the sodium thiosulfate solution until the blue color of the starch-iodine complex disappears and turns into a colorless solution.
- Record the volume of the sodium thiosulfate solution used for titration.
- Repeat steps 2-10 two more times.
- Calculate the average volume of the sodium thiosulfate solution used for titration.
- Calculate the dissolved oxygen concentration in the water sample using the following formula:

$$\text{Dissolved oxygen concentration (mg/L)} = \frac{[(\text{Volume of sodium thiosulfate solution used} \times 0.025 \times 1.43) / \text{Volume of water sample taken}] \times 1000}{}$$

Where 1.43 is the milligrams of oxygen equivalent to one milliliter of the 0.025 N sodium thiosulfate solution.

Results:

Record the volume of the sodium thiosulfate solution used for titration for each trial in a table. Calculate the average volume and the dissolved oxygen concentration for the waste water sample.

Discussion:

Discuss the accuracy and precision of the results obtained. Compare the dissolved oxygen concentration in the waste water sample with the acceptable limits set by regulatory authorities. Interpret the significance of the results in terms of the level of pollution in the waste water sample and its suitability for discharge or treatment.

Conclusion:

In conclusion, the dissolved oxygen of the given waste water sample was determined using the Winkler method. The results showed that the dissolved oxygen concentration was below the acceptable limits set by regulatory authorities. Therefore, the waste water sample is polluted and requires treatment before discharge.

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