17CVCC87- ENVIRONMENTAL ENGINEERING LABORATORY

(SCBCS-2017 Regulation)

DEPARTMENT OF CIVIL ENGINEERING

LAB MANUAL

HoD/Civil Engg.

17CVCC87- ENVIRONMENTAL ENGINEERING LABORATORY

OBJECTIVE

The students completing the course will be able to characterize wastewater and conduct treatability studies. To expected to be aware of the procedure for quantifying quality parameters for water and sewage. To be conducted for characterization of water and municipal sewage

LIST OF EXPERIMENTS

1. Sampling, preservation methods and significance of characterisation of water and waste water.
2. Determination of
   i) pH and turbidity
   ii) Hardness
3. Determination of Iron & Fluoride
4. Determination of residual Chlorine
5. Determination of Chlorides
6. Determination of Ammonia Nitrogen
7. Determination of Sulphate
8. Determination of Optimum Coagulant Dosage
9. Determination of available Chlorine in Bleaching powder
10. Determination of Dissolved Oxygen
11. Determination of suspended, volatile and fixed solids
12. B.O.D. test
13. C.O.D. test
14. Introduction to Bacteriological Analysis (Demonstration only)

TOTAL 45 PERIODS

REFERENCES

1.ENVIRONMENTAL ENGINEERING LAB MANUAL by VMKV Engineering College

2.Standard methods for the examination of water and wastewater, APHA, 20th Edition, Washington, 1998

3. Garg, S.K., "Environmental Engineering Vol. I & II", Khanna Publishers, New Delhi, 199

LIST OF EQUIPMENT
(For a batch of 30 students)

1. pH meter - 1 no.
2. Turbidity meter - 1 no.
3. Conductivity meter - 1 No.
4. Refrigerator - 1 No.
5. BOD incubator - 1 No.
6. Muffle furnace - 1 No.
7. Hot air oven - 1 No.
8. Magnetic stirrer with hot plates - 5 Nos.
9. Desiccator - 1 No.
10. Jar test apparatus - 1 No.
11. Water bath - 1 No.
12. Furniture - 1 lot
13. Glass waves / Crucibles - 1 lot
14. Chemicals - 1 lot
15. COD apparatus - 1 No.
16. Kjeldane apparatus - 1 No.
17. Heating mantles - 5 Nos.
18. Calorimeter - 1 No.
19. Chlorine comparator - 1 No.
20. Furniture Work table - 10 Nos.
21. Beaker - 30 Nos.
22. Standard flask - 30 Nos.
23. Burette with stand - 15 Nos.
24. Pipette - 15 Nos.
25. Crucible - 15 Nos.
26. Filtration assembly - 1 No.
27. Chemicals – Lot

LIST OF EXPERIMENTS

SAMPLING, PRESERVATION METHODS AND SIGNIFICANCE OF CHARACTERISATION OF WATER AND WASTEWATER.

Exp No: 1
Date

Aim

To study about the sampling, preservation methods and significance of characterization of water and wastewater.

Sampling

Random samples or grab samples are mostly collected manually from pipes, reservoirs, rivers, streams or drains. However, random samples can also be taken automatically. Each sample provides data on waste water quality at the collection time of sampling and cannot represent average conditions. Hence it should not be used as a basis for design parameter of treatment.

Grab samples are useful in determining the effects of extreme conditions of the waste during the time composite samples are being collected or when the waste water flow is intermittent. Composite samples or pooled samples can be obtained from an automatic sampler according to times or quantities. Such samples enable conclusions to be drawn about the water quality over longer periods. However, in most cases, peak concentrations cannot be identified in such pooled samples.

In quantity proportional sampling, volumes proportional to flow are collected at constant time intervals. Where the flow is continuously monitored, an almost quantity proportional pooled sample can be prepared by mixing relevant amounts from various single samples.

Sampling techniques

1. Topographic and specialized maps
2. Aerial photographs
3. Waterway plans, drainage plan

Preservation

During sampling, the water is removed from its natural environment. Due to this change, the chemical composition of water may not remain same, but may tend to adjust itself according to its new environment. The contents of water samples can alter at very different rates. These steps allow tests to be carried out even after longer period of time have elapsed.

Many ingredients are so stable that no special precaution for transport and storage are necessary. These are mainly the inorganic components. Water which are absolutely free of organics and micro-organisms show no microbiological degradation processes. The physic-chemical changes possible, where large amounts of organics materials are present and the conditions are suitable for growth of micro-organisms, the water contents can change in a very short time.

The following changes are possible in water samples

(i) Oxidation of components via dissolved oxygen
(ii) Precipitation and co-precipitation of inorganics through milieu changes (CaCO3, metal hydroxides)
(iii) Adsorption of dissolved trace components on the container walls.
(iv) Changes in parameters as a result of microbiological activity (e.g. pH value, oxygen, carbondioxide, BOD, trace organics).

General Information

In water and wastewater analysis, the results are usually reported in terms of mg/L of some particular ion, element or compound. It is most convenient to have the standard titrating agent of such strength, that 1mL is equivalent to 1mg of material being measured. Thus 1 litre of the standard solution is usually equivalent to 1g of the standard substance.

Normality

The desired normality of the titrant is obtained by the relationship of 1 to the equivalent weight of the measured material. Thus normality of acid solution to measure ammonia, ammonia nitrogen, and alkalinity as CaCO3

Ammonia — 1/eq. wt. = 1/17 = N/17 = 0.0588N
Ammonia N — 1/eq. wt. = 1/14 = N/14 = 0.0715N
Alkalinity — 1/eq. wt. = 1/50 = N/50 = 0.020N

The normality of basic solution to measure mineral acidity as CaCO3 is

Acidity — 1/eq. wt. = 1/50 = N/50 = 0.020N

The normality of silver nitrate to measure chloride and sodium chloride is

Chloride — 1/eq. wt. = 1/35.45 = N/35.45 = 0.0282N
Sodium chloride — 1/eq. wt. = 1/58.44 = N/58.44 = 0.071N

Thus the substance measured is calculated as follows

$$= \frac{\text{mL of titrant used} \times 1,000}{\text{mL of sample}} \text{ mg/L}$$

Most materials subjected to the analysis of water and wastewater fall in the realm of dilute solutions i.e., a few mg in a litre. So the results are normally expressed in mg/L or ppm. Parts per million (ppm) is a weight ratio; but mg/L is a weight by volume ratio. The relationship is given as follows

$$\text{ppm} = \frac{\text{mg/L}}{\text{Sp.gr.}}$$

If concentrations are less than 0.1 mg /L, express them in µg/L (micrograms per litre). If concentrations are more than 10,000 mg/L, they are expressed in percentages.

Plotting of Graphs

Rules listed by Worthing and Geffner are to be followed while plotting graphs.
They are

1. The independent and dependent variables should be plotted on abscissa and ordinate respectively.

2. The scale should be so chosen that the value of either coordinate could be found quickly and easily.
3. The curve should cover as much of the graph sheet as possible.
4. The scales should be so chosen that the slope of the curve approach unity as nearly as possible.
5. The variables should be chosen to give a plot that will be as nearly a straight line as possible.

STANDARDS OF WATER QUALITY

Standards of water quality are presented as follows

Bacteriological Quality

1. Treated water In 90% of the samples examined throughout the year, the coliform bacteria shall not be detected or the MPN index shall be less than 10. None of the samples shall have an MPN index of coliform bacteria in excess of 10. An MPN index of 8–10 shall not occur in consecutive samples.

2. Untreated water In 90% of the samples examined throughout the year, the MPN index of coliform organisms should not be less than 10. None of the samples should show an MPN index greater than 20. An MPN index of 15 or more should not be permitted in consecutive samples.

Chemical and Physical Quality

Classification	*Substances*	*Maximum allowable concentration*
1. Toxic substances	Lead (Pb)	0.1 mg/L
	Selenium (Se)	0.05 mg/L
	Arsenic (As)	0.2 mg/L
	Chromium (Cr^{6+})	0.05 mg/L
	Cyanide (CN)	0.01 mg/L
2. Chemical substances which may affect health	Fluoride (F^-)	0.08–1.0 mg/L
	Nitrate (NO_3^-)	50.0 mg/L
	Total solids	500 mg/L
	Colour	5 Units
	Turbidity	5 Units
	Taste	Unobjectionable
	Odour	Unobjectionable
	Manganese (Mn)	0.1 mg/L

Contd...

3. Chemical substances affecting the potability of water	Iron (Fe)	0.3 mg/L
	Copper (Cu)	1.0 mg/L
	Zinc (Zn)	5.0 mg/L
	Calcium (Ca)	75 mg/L
	Magnesium (Mg)	50 mg/L
	Sulphate	200 mg/L
	Chloride (Cl^-)	200 mg/L
	pH range	7.0–8.5
	Phenolic substances	0.001mg/L

Significance, characterization and determination of Chemical Parameters

Chemical parameters and their significance are presented as follows. The methods of the analysis adopted are also presented. However, only simple methods will be dealt within this manual.

Chemical parameters commonly determined in natural waters and water supplies

No.	*Chemical species*	*Significance in water*	*Methods of analysis commonly used*
1.	Acidity	Indicative of industrial pollution, acid mine drainage	Titration
2.	Alkalinity	Water treatment, buffering, algal productivity	Titration
3.	Ammonia	Productivity, pollution	Colorimetry
4.	Calcium	Hardness, productivity treatment	Atomic absorption
5.	Carbon dioxide	Bacterial action, corrosion	Titration, calculation
6.	Chloride	Saline water contamination	Titration, potentiometry
7.	Chlorine	Water treatment	Colorimetry, titration
8.	Fluoride	Water treatment, toxic at high level	Colorimetry, potentiometry
9.	Hardness	Water quality, treatment	Titration, atomic absorption
10.	Iron	Water quality, treatment	Colorimetry, atomic absorption
11.	Magnesium	Hardness	Atomic absorption
12.	Manganese	Water quality	Atomic absorption
13.	Nitrate	Productivity, toxicity	Colorimetry, potentiometry
14.	Nitrite	Toxic, pollutant	Colorimetry

15.	Nitrogen (albumin.)	Proteinaceous material	Colorimetry
16.	Nitrogen (organic)	Organic pollution	Colorimetry
17.	Oxygen	Water quality	Titration, electrochemical
18.	BOD	Water quality, pollution	Microbiological titration
19.	COD	Water quality, pollution	Chemical oxidation-reduction
20.	pH	Water quality, pollution	Potentiometry
21.	Phosphate	Water quality, pollution	Colorimetry
22.	Sulphate	Water quality, pollution	Gravimetry, turbidimetry
23.	Sulphide	Water quality, pollution	Colorimetry, potentiometric titration

DETERMINATION OF TURBIDITY

Exp No: 2
Date

Aim

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

Principle

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

Apparatus required

Nephelometer with accessories

Reagents

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

Preparation of Turbidity Free Distilled Water

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

Preparation of Formazine Turbidity Concentrate

(a) Solution I

Weigh accurately 5 g of 'Anal–R' quality hydrazine sulphate $(NH2)2H2SO4$ into a 500 mL volumetric flask and add distilled water to make up to the mark. Leave the mixture to stand for 4 hours.

(b) Solution II

Weigh accurately 50g of 'Anal–R' quality hexamethylene tetramine $(CH2)6N4$ (hexamine) into a 500 Ml volumetric flask and add distilled water to make up to the mark.

Mix equal volume of solution I and II to form formazine turbidity concentrate. Allow it to stand in a closed container at 25°C to 30°C for 48 hours to produce insoluble white turbidity corresponding to 4000 NTU.

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

Preparation of Formazine Standard

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

Note Formazine standard 100 NTU should be prepared weekly.

Procedure

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

Measurement of turbidity less than 100 NTU

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

Measurement of turbidity above 100 NTU

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

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

where,
A = NTU found in diluted sample
B = volume of dilution water in mL
C = sample volume taken for dilution in mL

Observation

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

Results

Description of sample	Turbidity in NTU

DETERMINATION OF pH

Exp No: 3
Date

Aim

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

Principle

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

Apparatus

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

Reagents

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

Procedure

(a) Using Universal Indicator

1. 10 mL of sample is taken in a cuvette.
2. Another 10 mL sample is taken in another cuvette and 0.2 mL of universal indicator is added and placed in the hole provided for.
3. A colour disc corresponding to this indicator is inserted into the comparator and the disc rotated such that the 2 circles indicate identical colours.
4. The reading is noted.

5. The procedure can be repeated using an indicator whose range is near the value obtained.
6. The exact pH is obtained.

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

(b) Using pH Papers

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

(c) Using pH Meter

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

Results

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

DETERMINATION OF HARDNESS

Exp No: 4
Date

Aim

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

Principle

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

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

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

Apparatus

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

Reagents

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

Procedure

1. Dilute 25 mL of sample (V) to about 50 mL with distilled water in an Erlenmeyer flask.
2. Add 1 mL of buffer solution.
3. Add two drops of indicator solution. The solution turns wine red in colour.
4. Add the standard EDTA titrant slowly with continuous stirring until the last reddish tinge disappears from the solution. The colour of the solution at the end point is blue under normal conditions.
5. Note down the volume of EDTA added (V1).

Observation

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

Calculation

$$\text{Hardness as } CaCO_3 = \frac{V_1 \times S \times 1000}{V} \text{ mg/L}$$

where, S = mg $CaCO_3$ equivalent to 1 mL of EDTA titrant

= 1 mg $CaCO_3$

$$\text{Hardness as } CaCO_3 = \frac{1000\ V_1}{V} = \ldots\ldots \text{ mg/L}$$

Results

Total hardness in mg/l as $CaCO_3$ ----------

DETERMINATION OF IRON AND FLUORIDE

Exp No: 5
Date

Aim

To determine the quantity of iron and fluoride present in the given sample of water.

Principle

Iron is usually present in natural water and is not objectionable, if concentration is less than 0.3 ppm. It may be in true solution in colloidal state that may be peptized by organic matter, in the inorganic and organic iron complexes, or in relatively coarse suspended particles. It may be either ferrous or ferric, suspended or filterable. Iron exists in soils and minerals mainly as insoluble ferric oxide and iron sulphide (pyrite). It occurs in some areas, also as ferrous carbonate (siderite), which is very slightly soluble.

The phenanthroline method is the preferred standard procedure for the measurement of iron in water except when phosphate or heavy metal interferences are present. The method depends upon the fact that 1, 10-phenanthroline combine with Fe++ to form an orange-red complex. Its colour conforms to Beer's law and is readily measured by visual or photometric comparison. Small concentration of iron can be most satisfactorily determined by colorimetric analysis. It is also based on Beer's law. By measuring the intensities of transmitted and incident light through a coloured solution and knowing its optical density or transmission, we can prepare a calibration curve and subsequent concentration can be read.

Phenanthroline Method

Apparatus required

1. Colorimetric equipment; one of the following is required
(a) Spectrophotometer, for use at 510 nm, providing a light path of 1 cm or longer.
(b) Nessler tubes, matched, 100 ml, tall form.
2. Glassware like conical flasks, pipettes and glass beads.

Reagents

1. Hydrochloric acid 2. Hydroxylamine solution
3. Ammonium acetate buffer solution 4. Sodium acetate solution
5. Phenanthroline solution 6. Stock iron solution
7. Standard iron solution (1 ml = 10 mg Fe)

Procedure

1. Pipette 10, 20, 30 and 50 mL. Standard iron solution into 100 mL conical flasks.
2. Add 1 mL hydroxylamine solution and 1 mL sodium acetate solution to each flask.
3. Dilute each to about 75 mL with distilled water.

4. Add 10 mL phenanthroline solution to each flask.
5. Make up the contents of each flask exactly to 100mL by adding distilled water and left stand for 10 minutes.
6. Take 50 mL distilled water in another conical flask.
7. Repeat steps 2 to 5 described above.
8. Measure the absorbance of each solution in a spectrophotometer at 508 nm against the reference blank prepared by treating distilled water as described in steps 6 and 7. Prepare a calibration graph taking meter reading on y-axis and concentration of iron on x-axis.
9. For visual comparison, pour the solution in 100 mL tall form Nessler tubes and keep them in a stand.
10. Mix the sample thoroughly and measure 50 mL into a conical flask.
11. Add 2 mL conc. hydrochloric acid (HCl) and 1mL hydroxylamine solution. Add a few glass beads and heat to boiling. To ensure dissolution of all the iron, continue boiling until the volume is reduced to 15 to 20 mL.
12. Cool the flask to room temperature and transfer the solution to a 100 mL Nessler tube.
13. Add 10 mL ammonium acetate buffer solution and 2 mL phenanthroline solution and dilute to the 100 mL mark with distilled water.
17. Mix thoroughly and allow at least 10 to 15 minutes for maximum colour development.
18. Measure the absorbance of the solution in a 1cm cell in a spectrophotometer at 508 nm.
19. Read off the conc. of iron (mg Fe) from the calibration graph for the corresponding meter reading.
20. For visual comparison, match the colour of the sample with that of the standard prepared in steps 1 to 7 above.
21. The matching colour standard will give the concentration of iron in the sample (µg Fe).

Result

Iron content of the sample (mg/l) = ----------

Fluoride content of the sample (mg/l) = ----------

DETERMINATION OF RESIDUAL CHLORINE

Exp No: 6
Date

i) By orthotolidine test procedure

Aim

To determine the residual chlorine in the water sample by orthotolidine test procedure using chloroscope.

Apparatus Required

New water chloroscope.

Reagents

Orthotolidine reagent

Chloroscope

It is a simple, study compact, portable and easy to operate. Built in convenient test tube holders, permeable top lid. Two glass test tubes. Left one for the untreated water (blank) and right one for the treated water.

The left side row of five standard filter discs varied colour density to compare the colour developed in the treated water after adding reagent looking through the right side row of inspection windows.

Procedure

1. Remove the top lid fill the left side test tube with untreated water and inject on the colour disc side.

2. Fill the right side test tube with treated water with chlorine solution or tablet and add 3 drops of orthotilidine reagent and shake well and then place it on right side.

3. Choose the top cover and view against light.

4. Compare the colour of water being test within the standard colour discs.

5. Note the reading of the matching disc. This values indicates the residual chlorine in ppm in the treated water.

6. The recommended permissible value is between 0.2 to 1.0 ppm.

Result

Observed value from disc comparator is ----------

ii) By titration method

Aim

To determine the residual chlorine in the given water sample by titration method.

Apparatus Required

1. Burette
2. Conical flask
3. Measuring jar

Principle

Chlorine is primarily added to the water for destroying the harmful micro organisms. Presence of excess chlorine intensifies the taste and odour of any other compounds in combination with ammonia.

Chlorine is a strong oxidizing agent and liberates iodine from potassium iodide. The liberated iodine is equivalent to the amount of chlorine and can be titrated against sodium thiosulphate using starch as an indicator.

Reagents

1. Acetic acid
2. Potassium iodide
3. Sodium thio sulphate 0.025N
4. Starch indicator

Procedure

1. Take 100ml of sample in a conical flask and add 5ml acetic acid. The point after the addition of acetic acid should be between 3 and 4.
2. Add approximate 1gm of KI crystal and mix with a stringing rod for about 15 minutes keeping it away from the direct sunlight.
3. Add a few drops of starch indicator and titrate against 0.02N sodium thio sulphate until the contents turn colourless from blue.

Tabulations

Burette solution sodium thio sulphate
Pipette solution chlorinated water sample
Indicator starch
End point blue to colourless.

S.NO	Vol of sample (V1 ml)	Burette Reading (ml)		Vol of sodium thio sulphate (V2 ml)	Concordant Value (ml)	Indicator
		Initial	Final			
1.						
2.						

Calculation

$$\text{Chloride in mg/Lt} = \frac{(\text{ml of } AgNO_3 \times \text{Normality of } AgNO_3 \times 1000 \times \text{Equivalent weight of Chlorine})}{\text{Volume of Sample}}$$

Result

The amount of residual chlorine present in the given sample of water is --------

DETERMINATION OF CHLORIDES

Exp No: 7
Date

Aim

To find the amount of chlorides present in the given water sample.

Apparatus Required

1. Burette
2. Conical flask
3. Measuring jar

Principle

Silver nitrate react with chlorine to form very slightly soluble white precipitate of Agcl at the end point when all the chlorides get precipitate free silver ions react with chromate to form reddish brown colour.

Reagents

1. Silver nitrate 0.01 N
2. Potassium chromate

Procedure

1. Take 20ml of the sample in a conical flask and add 1-5 ml of K_2CrO_4 solution.

2. Titrate the contents against 0.01N $AgNO_3$ untill a red tinge colour appears.

Tabulations

Burette solution $AgNO_3$
Pipette solution water sample
Indicator K_2CrO_4
End point Yellow to red tinge colour.

S.NO	VOL OF Nacl(V1 ml)	BURETTE READING (ml)		VOL OF $AgNO_3$ (V2 ml)	CONCORDANT VALUE (ml)	INDICATOR
		Initial	Final			
1.						
2.						

Calculation

$$\text{Chloride in mg/Lt} = \frac{(\text{ml of } AgNO_3 \times \text{Normality of } AgNO_3 \times 1000 \times \text{Molecular weight of Chlorine})}{\text{Volume of Sample}}$$

Result

The amount of chloride present in the given sample is ----------

DETERMINATION OF AMMONIA NITROGEN

Exp No: 8
Date

Aim

To determine the amount of Ammonia Nitrogen present in the given sample.

Principle

Ammonium ion reacts with Nessler's reagent (K_2HgI_4) to form a brown colour substance, and can be determined colorimentically. Most of the natural waters and wastewaters have interfering substances, therefore, the steam distillation of ammonia becomes essential.

Apparatus required

1. Measuring jar
2. Conical flask
3. Burette
4. Pipette

Reagents

1. Phosphate buffer solution
2. Boric acid
3. Methyl orange indicator
4. Sulphuric acid 0.02N

Procedure

1. Take 50ml of the sample in a conical flask.
2. Add 5ml of phosphate buffer solution and 10 ml of boric acid solution.
3. Add 3 -5 drops of methyl orange indicator.
4. Titrate against 0.02N of sulphuric acid till the end point is changes from orange to yellow.

Calculation

$$NH_3\text{-}N_2 \text{ mg/l} = \frac{\text{ml of } H_2SO_4 \times 0.28 \times 1000}{\text{Ml of sample}}$$

Tabulation

S.No	Vol of water sample (ml)	Initial burette reading (ml)	Final burette reading (ml)	Concurrent burette reading (ml)	Vol of sulphuric acid (ml)

Result

The amount of Ammonia Nitrogen present in the given sample is---------- mg/l.

DETERMINATION OF SULPHATE

Exp No: 9
Date

Aim

To find the sulphates in the given sample.

Principle

Benzidine hydorochloride reacts with sulphates in HCl solution to form a slightly soluble compound of benzidine sulphuric acid.

$CaSO_4 + C_{12}H_8 (NH_2)_2.2HCl$ ------ $C_{12} H_8(NH_2)_2 H_2SO_4 + Ca^{++} + Mg^{++} + Cl_2$

The compound is filtered and washed entirely free of HCl. The amount of H2SO4 in the compound is determined by titration with standard NaOH (0.05N)

$C_{12} H_8(NH_2)_2 H_2SO_4 + 2NaOH$ -------$Na_2SO_4 + C_{12}H_8(Na_2)_2 2H_2O$

Apparatus Required

Filter paper
Beaker
Hot pan
Burette
Pipettes

Reagents

Hydroxylamine chloride
Benzidine hydrochloride
NaOH (0.05N)
Phenolpthalein indicator

Procedure

1. Take 125ml of sample in a 400ml beaker.
2. add 5ml of hydroxylamine chloride and then add 10ml benzidine hydrochloride.
3. stir the mixture vigorously and allow the precipitate to settle.
4. Filter the solution and wash the beaker and the filter paper with cold distilled water.
5. pierce the filter paper in the funnel and wash the precipitate formed on the filter paper to the original beaker with 100 to 150ml distilled water.
6. heat the beaker to dissolve the contents for 20 to 30 minutes.
7. add 2 drops of phenolphthalein indicator and titrate with 0.05N NaOH until pink colour is developed.

Calculations

Concentration of sulphates (mg/l) = Vol. of 0.05N NaOH x 38.4

Vol. of the sample taken

Observations

Sample details	Vol of sample taken (ml)	Initial burette reading (ml)	Final burette reading (ml)	vol of NaOH solution used (ml)	Sulphates (mg/l)

Results

The sulphate present in the sample is ----------

DETERMINATION OF OPTIMUM COAGULANT DOSAGE

Exp No: 10
Date

Aim

To find out the optimum coagulant required to precipitate turbid particles present in the water.

Principle

Metal salts hydrolyse in presence of the natural alkalinity to form metal hydroxides. The divalent cations can reduce the zeta-potential, while the metal hydroxides are good absorbents and hence remove the suspended particles by enmeshing them.

Apparatus Required

1. Jars mixer
2. Turbid water
3. Beakers
4. Pipettes
5. Turbidity meter
6. pH meter

Reagents

Alum solution

Procedure

1. 200ml of water sample is taken in each jar. Increasing dose of alum (1%) i.e. 1gm/100ml of distilled water added to slowly for 15min and allowed to stand for 15min.

2. The jars are observed and the settling of sediments are noted. The quality of alum added to the jar containing the clearest solution is noted.

3. Take the sample out of beaker and test for turbidity in each trial plot the curve on x and y axis of the graph sheet. Take the alum dosage in ml along x axis and turbidity along y axis.

Observations

Raw water turbidity (NTU) =

Raw water pH =

Raw water alkalinity (mg/l) =

Sample details	Dosage of coagulant	Residual turbidity	pH	Alkalinity mg/l

Result

The optimum dosage of coagulant required to remove turbidity in the given water sample is…….

DETERMINATION OF AVAILABLE CHLORINE IN BLEACHING POWDER

Exp No: 11
Date

Aim

To determine the available chlorine in the given bleaching powder.

Principle

Chlorine is a strong oxidizing agent and liberates iodine from iodine in

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

Starch gives blue colour with iodine

I_2+starch--→Blue Colour

The liberated iodine is liberated against standard sodium thiosulphate reducing agent.

$I_2+2Na_2S_2O_2 \rightarrow Na_2S_4O_6 + 2Nal$

The disappearance of blue colour indicator the completion of reaction with free iodine converted back to iodine.

Apparatus Required

1. Conical flask
2. Burette,Pipette

Reagents

1. Concentrated acetic acid
2. Potassium iodine crystals
3. Sodium thiosulphate(0.025N)
4. Starch indicator

Procedure

1. 5 gm of fresh bleaching powder is taken and is added to a small quantity of water and made into fine paste some more is added stired well and allowed to settle for a few minutes.
2. 25ml of bleaching powder solution is taken in a conical flask and pinch of potassium iodine is added.
3. 10 ml of acetic acid is added and allow for the reaction to complete.
4. 1ml of starch solution is added and is titration is continued till the disappearing blue colour.
5. The sample is titrated against sodium thiosulphate solution until yellow colour of liberated iodine fades.
6. The quantity of $Na_2S_4O_6$ added noted down (V1).
7. Same procedure is repeated for distilled water.
8. The quantity of $Na_2S_4O_6$ consumed is noted down (V2).

Calculations

Concentration of chlorine = $\frac{(V1 - V2) \times N \times 35.45 \times 1000}{\text{Vol of bleaching powder solution}}$

1gm of bleaching powder contains ---------- mg of chlorine.

Percentage of chlorine content in bleaching powder = ----------

Observations

Sample details	Vol of sample taken (ml)	Initial burette reading (ml)	Final burette reading (ml)	Available chlorine (mg/l)	% of chlorine

Result

The available chlorine in given sample of bleaching powder=…….

Percentage of chlorine content in bleaching powder=……

DETERMINATION OF DISSOLVED OXYGEN

Exp No: 12
Date

Aim

To determine the amount of dissolved oxygen present in the given water sample.

Principle

The magnesium sulphate react with aloud(KOH) a form a white precipitate of magneous hydroxide which in the present of oxygen gets oxidized method magnese ions are reduced by iodide ions which gets converted into iodine equivalent to the original concentration of oxygen in the sample.

Apparatus Required

1. Burette
2. Conical flask
3. BOD bottle
4. Measuring jar

Reagents

1. Sodium thiosulphate-0.01N
2. Alkaline potassium iodine solution-2ml
3. Magneous sulphate solution-2ml
4. Starch indicator
5. Sulphuric acid

Procedure

1. 1.Fill the sample in a glass stoppered bottle to known 150ml carefully avoiding and kind of bubbling and trapping of the air bubbles in the bottle after placing the stopper.
2. Add 2ml each $MnSO_4$ and alkaline K_2SO_4 solution of well below the surface from the walls.The reagents can also be powered at the bottom of the bottle with the help of specisl pipette ringed to ensure better mixing of reagents with the sample.
3. Place the stopper and shake the contents well by inversting the bottle presently keep the bottle for same to settle down the precipitable if the titration is to be prolonged for few days keep the sample at this stage with the precipitate add 2ml of concentrated H_2SO_4 and shake well to dissolve the precipitate.
4. Remove either whole contents of a part of the take 100ml in a conical flask for filtration present and bubbling to avoid further mixing of oxygen.
5. Titrate the contents within one hour of dissolution of precipitate against sodium this sulphate solution using starch and indicator.

Calculations

1ml of 0.025N $Na_2S_2O_3$ is equivalent to 0.2 mg of O_2 since the volume of the sample is 200 mg.

1ml of sodium thio sulphate is equivalent to $\frac{(0.2 \times 1000)}{200}$ mg/l = 1mg/l

Observations

Sample details	Temp. of sample oC	Vol of sample taken ml	Initial burette reading ml	Final burette reading ml	Ml of $Na_2S_2O_3$ solution used	DO in mg/l

Result

The amount of dissolved oxygen present in the given water sample is……..

DETERMINATION OF SUSPENDED, VOLATILE AND FIXED SOLIDS

Exp No: 13
Date

Aim

To determine the amount of total solids, suspended solids and dissolved solids in given sample.

Apparatus Required

1. Muffle furnace
2. Hot plate or water bath oven
3. Evaporating dish

Principle

Residual after the evaporation and subsequent drying in oven at specific temperature (103^o to 105^o C) of a known volume of sample is called as total solids**.**

The suspended solids can be found by filtering the water sample and weighing the residue left on the filter paper.

Procedure

1. Take an evaporating disc of at least 100ml capacity liquid at 550 to 50^o in a muffle furnace for about an hour cool in a desicator and weigh it as W1.
2. Evaporate 10 ml of unfiltered sample few more in the case the solids are less than 250mg/lt in evaporating dish on a water bath over list plate lowing temperature are not more than 98^oC.
3. Cool in a desicator and weigh the residue left in a dish.
4. Weigh the filter paper without any moisture (W5)
5. Dry the paper in the oven then weigh the residue left in paper (W4).

CALCULATIONS

$$\text{Total solids (mg/l)} = \frac{(W2-W1) \times 1000}{V}$$

$$\text{Suspended Solids (mg/l)} = \frac{(W4 - W3) \times 1000}{V}$$

Dissolved Solids = Total solids – Suspended solids

Result

Total Solids =---------- mg/l
Suspended solids = ---------- mg/l
Dissolved solids = ----------mg/l

B.O.D TEST

Exp No: 14
Date

Aim

To determine the BOD in the given wastewater sample.

Apparatus Required

BOD incubator
BOD bottle (300ml)

Principle

If sufficient oxygen is available in wastewater, the useful aerobic bacteria will flourish and cause the aerobic biological decomposition of wastewater which will continue until oxidation is completed.

The amount of oxygen consumed in this process is the BOD. Polluted waters will continue to absorb oxygen for many months, and it is not practically feasible to determine this ultimate oxygen demand.

Reagents

1. Distilled water
2. Phosphate buffer solution
3. Magnesium sulphate solution
4. Calcium chloride solution
5. Ferric chloride solution
6. Sodium thiosulphate solution

Procedure

1. Mix a known volume (4ml) of a sample of wastewater with a known volume of aerated pure water to make 300ml diluted sample and then calculate the D.O.of this diluted sample.
2. The diluted sample is then incubated for 5 days at 20^{o} C .
3. The D.O.of the diluted sample ,after this period of incubation, is again calculated.
4. The difference between the initial D.O. value and the final D.O. value will indicate the oxygen consumed by the sewage in 5 days .

CALCULATION

BOD5=D.O.consumed by the diluted sample x (volume of diluted sample)

Volume of undiluted sewage

= dilution factor

Observations

S.No	Vol of sample (ml)	Dilution ratio	Initial DO of sample mg/l	Final DO of sample mg/l	Initial DO of Blank mg/l	Final DO of blank mg/l	5 days BOD at 20^oC mg/l

Result

BOD_5 of given sample at 20^oC in mg/l =

C.O.D TEST

Exp No: 15
Date

Aim

To find the chemical oxygen demand of given wastewater sample.

Principle

The organic matter present in sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of $H2SO_4$ to produce CO_2 and H_2O. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with $Fe(NH_4)_2(SO_4)_2$. The dichromate consumed gives the O_2 required to oxidation of the organic matter**.**

Apparatus Required

1. Reflux apparatus
2. Hot plate/heating mantle
3. Burette

Reagents

1. Standard potassium dichromate 0.25N
2. Sulphuric acid with reagent (Conc.H_2SO_4 + Ag_2SO_4)
3. Standard ferrous ammonium sulphate 0.1N
4. Ferroin indicator
5. Mercuric sulphate

Procedure

1. Place 0.4gm of $HgSO_4$ in the reflux flask.
2. Add 20ml of sample (or an aliquot diluted to 20ml)
3. 10ml of more concentrated dichromate solution are placed into flask together with glass beeds.
4. Add slowly 30ml of H_2SO_4 containing Ag_2SO_4 and mix thoroughly.
5. Connect the flask to condenser. Mix the contents thoroughly before heating.
6. Improper mixing results in bumping and the sample may be blown out.
7. Reflux for a minimum period of 2 hours. Cool and wash down the condenser with distilled water.
8. Dilute the sample to make up 150ml and cool.
9. Titrate excess $K_2Cr_2O_7$ with 0.1N $Fe(NH_4)_2SO_4$ using ferroin indicator.
10. Sharp colour change from blue green to wine red indicates the end point.
11. Reflux the blank in the same manner using distilled water instead of sample.

Calculations

Quantity of $Fe(NH_4)_2SO_4$ added for blank = A ml
Quantity of $Fe(NH_4)_2SO_4$ added for the sample = B ml

$$COD = \frac{(A - B) \times \text{normality of } Fe(NH_4)_2SO_4 \times 8 \times 1000}{\text{Quantity of sample (ml)}}$$

Observations

S.NO	Vol of sample (ml)	Burette Reading (ml)		Vol of $Fe(NH_4)_2SO_4$ (ml)	COD of the sample mg/l	INDICATOR
		Initial	Final			
1.						
2.						

Result

The COD of the given sample is ----------

INTRODUCTION TO BACTERIOLOGICAL ANALYSIS

Exp No: 16
Date

Aim

To find out total bacterial count present in a given sample.

Apparatus required

1. Incubator
2. Petri dishes
3. Conical flask
4. Thermometer with precision 0.1oC
5. Pipettes
6. Spirit lamp
7. Colony counter
8. Water bath

Reagents

1. Nutrient agar medium
2. Sterile distilled water

Procedure

1. Select the dilution ratio's depend on expected total bacterial count (10, 1100, 11000)

2. A separate sterile pipette should be used for each dilution.

3. Transfer 1ml from undiluted and 0.1ml and 0.01ml from diluted samples in sterile petric dish. After delivering the sample, touch the tip of pipette to a dry spot in the plates.

4. Pour 10ml of sterile nutrient agar medium of the temperature 44-46oC to these petri dishes by gently opening dish plates slightly.

5. Mix the medium thoroughly with sample in petri plates. When the media is solidified invert the plates and keep for incubations at $37^{o}C$ +_ $0.5^{o}C$ for 44+_ 4 hrous.

6. The visible colonies are then counted with the aid of a 6-8 x magnify glass or colony counter.

Observations

Dilution	No of colonies per dish	SPC/ml	Mean SPC/ml
10 1100 (dilution factor 100) 11000 (dilution factor 1000)			

Calculations

SPC/ml = colonies counted

Dilution factor

Result

Mean SPC per ml is ----------

Environmental Significance

The total bacterial count is the number of visible colonies under a magnification of 6-8 x which have developed under defined conditions. It provides a measure of the degree of microbiological contamination of the water and especially of sudden bacterial invasions.

Its values are useful in warning about excessive microbial growth in any water and also in judging the efficiency of water and wastewater treatment in removing organisms.