

DEPARTMENT OF CIVIL ENGINEERING

Subject- Public Health Lab

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Ex No: 1

Study of sampling and preservation methods anti

Date:

Significance of characterisation of water and waste water

Aim

To study the sampling and preservation methods in water and waste water characterization and to learn the significance of characterisation of water and waste water

Sampling Programme and Procedures

The collection of a representative sample is the most important function of an environmentalist. The interpretation of results and recommendation for prevention and corrective treatment are all based on the analysis report. Scrupulous care in the collection of samples is therefore necessary to ensure that the sample is representative of the body of water under examination and to avoid spoilage and accidental contamination of the sample during collection and transport.

Methods of sampling

Three types of samples are often collected depending on situations

a. Grab Samples

Grab samples are samples collected at a designated place at a particular time. They represent the composition at the time and space. When a source is known to vary in time, as in the case of waste effluents, grab samples collected at various time intervals and analysed separately can be of greater value.

b. Composite samples

Composite samples are a mixture of grab samples collected at one sampling point at different times. Individual samples are collected in wide mouth bottles every hour and mixed in volume proportional to the flow. The composite values are useful for observing average values.

c. Integrated samples

Integrated samples are a mixture of grab samples collected from different points simultaneously and mixed in equal volumes. Individual samples are collected from both banks of a river and at varying depths to represent available situations.

Sampling and preservation Requirements:

1. Physical and Chemical Requirements:

For general physical and chemical examination, the sample should be collected in a chemically clean bottle made of good quality glass fitted with a ground glass stopper or a chemically inert polyethylene container. The volume of sample to be collected would depend on the selection of tests; however, for general examination 3.0 litre sample would be sufficient,

The following precautions must be taken while collecting the sample

- i) The sampling location is representative of the water body
- ii) The place is devoid of floating material

Where ever possible the sample should be collected 15cm, below the surface or as the situation warrants

No physical activity is permitted upstream of sampling point Shorter the time between collection and examination, the reliable will be the analytical results. For certain constituents and physical values, immediate analysis in the field is required, because, the composition of water may change before it arrives at the laboratory.

The maximum limits of storage are:

Unpolluted water: 72 hours

Slightly polluted. : 48 hours

Grossly polluted: 1 2hours

Some determinations are more likely to be affected by storage than others. Temperature may change, pH may change significantly, and dissolved gases may be evolved and lost (O₂, CO₂. and H₂S)

Frequency of sampling:

Frequency depends on objectives. Yet, collection of samples of both raw and treated waters should be carried out as frequently as possible and at least once in every three months. Some waters undergo more pronounced seasonal variation and therefore require more frequent testing. Samples from treatment units should be collected and analyses frequently, at least one from each unit daily.

2. Bacteriological requirements:

The samples for bacteriological examination are collected in sterilized, neutral glass, glass-stopper 80z, and 300 ml bottles. The stopper and the neck should be protected by paper or parchment cover. If the sample is likely to contain traces of residual chlorine, an amount equal to 3.0 mg of sodium thiosulfite ($\text{Na}_2\text{S}_2\text{O}_3$, 51120) to neutralize chlorine is added to the bottle before sterilization. The sterilization is done at 15 psi (121°C) for 20-30 minutes in an autoclave.

The sterilized sample bottle should be kept unopened until the time of collection. The stopper should be removed with care to eliminate chances of spoiling and contamination and should never be rinsed. After filling, the stopper should be replaced immediately. The place of collection should be predetermined and procedure of collection conditioned depending on the source.

The standard procedure in sampling from a water faucet or tap is as follows:

- a) Flame the tap briefly to kill clinging bacteria. This can be done with a piece of burning paper.
- b) Turn on the water and allow it to run for 1 min.
- c) Remove the stopper from the bottle, being careful not to touch the inner portions of the stopper or bottle neck.
- d) Fill bottle carefully, allowing no water to enter that has come in contact with hands. It is sometimes necessary to collect a sample from a reservoir or basin. If the water can be reached, remove the stopper, plunge the bottle below the surface and move the bottle while it is filling, so that no water will enter that has been in contact with hand. If the water is out of reach, as in a dug well, the bottle can be lowered with a cord.

The sample after collection should be examined immediately, preferably within one hour. If the conditions do not permit immediate examination, the sample should be stored at low temperatures. This period should in case be more than 24 hours. If storage or transportation is necessary, they should be kept at a temperature between 0°C and 10°C .

Frequency of sampling:

The frequency of sampling should be fixed depending on the magnitude of the problem involved. The number of samples to be examined from drinking water supply distribution system is normally decided on the basis of population served as given in the tabulation:

Population	Treated / untreated water entering distribution system	
	Max.interval between successive sampling	Max.no.of samples to be examined.
Upto 20,000	1 month	One sample for every 5000 population
20,001 - 50000	15 days	
50,001 - 1,00,000	4 days	
More than 100,000	1 day	One sample for every 10,000 population

The raw water should be examined as frequently as the situation demands. The frequency is also determined based on objectives of study.

3. Biological Requirements:

In general the samples for biological examination are collected in wide mouth, clean glass bottles of 2.0 litre capacity. They are never filled completely. This method is employed when total microscopic count is the aim. In some specific cases the concentrate of a sample may be collected through plankton nets made of bolting silk cloth, or the sample filtered through Sedge wick Rafter funnels.

In general the sample must be examined microscopically within one hour of collections. If the facilities do not permit an immediate examination, it should be preserved after collection by addition of 2 ml neutralized (pH 7.0) formaline to each 100 ml of the sample.

There is no practice about the frequency of sampling but the examination should be made regularly, or else as the situation demands. Benthos study is complex, Collection through cages placed at proper preselected sites for a defined period of time is recommended.

Preservation Methods for Characterisation of samples:

Determination	Containers	Minimum sample size ml	Preservation	Max.storage recommended
pH	P,G	-	Analyse immediately preferably in field	0.5 h
Solids	P,G	300	Refrigeration	7 d
Sulfates	P,G	100	Refrigerate	28 d
D.o	G,BOD Bottle	300	Titration may be delayed after fixation (1 ml Alk.KI and 1 ml MnSO ₄) and acidification	8 h
Turbidity	P,G	-	Analyse same day store in dark, refrigerate	24 h
Hardness	P,G	100	Add HNO ₃ to pH<2	6 months
Fluoride	P	300	None	28 d
God	P,G	100	Analyse as soon as possible or add H ₂ SO ₄ to pH<2	7 d
B.O.D	P,G	1000	Refrigerate	6h
Chlorine residuals	P,G	500	Analyse immediately	0.5 h
Ammoniacal Nitrogen	P,G	500	Analyse as soon as possible or add H ₂ SO ₄ refrigerate	6 months

Note: Refrigerate – Storage at 4₀C; P=plastic (Polyethylene or equivalent), G=Glass, neutral.

Significance of Characterisation of various parameters:

Natural waters are never completely pure. During their precipitation and passage over or through ground they require a wide variety of dissolved and suspended impurities. The concentrations of these impurities are seldom large in ordinary chemical sense but they modify the chemical behaviour of water or its usefulness.

The waste waters generally have values far higher than in waters.

Some of these impurities are toxic, some may affect health, and some affect the portability while others indicate pollution. A list of such impurities is given below:

Toxic substances	Max. allowable limit (W.H.O. Standards) mg/L
Lead	0.05
Arsenic	0.05
Selenium	0.01
Chromium	0.05
Cyanide	0.2
Cadmium	0.01
Radio active compounds (gross beta activity)	1000μc/L
Components hazardous to health	
Fluorides	1.5
Nitrates as NO₃	45
Compounds affecting the portability	
TDS	1500
Iron	5.0
Managese	5.0

Copper	1.5
Zinc	1.5
Magnesium plus - sodium sulfate	1000
Surfactants (ABS)	0.5
Chemical indicators of population	
BOD	60
COD	10.0
Total Nitrogen exclusive of NO₃	1.0
Ammonia cal Nitrogen as NH₃	0.5
Carbon chloroform extract (CCE)	0.5
Oil and Grease	1.0
D.O	40% saturation

Hardness

The study of hardness is important from the point of view of industrial utilization of water especially in boilers, where scales are formed. Hardness in municipal supplies increases the consumption of soap, fuel, tea leaves etc. in the household and renders it unsuitable for use in air-conditioning.

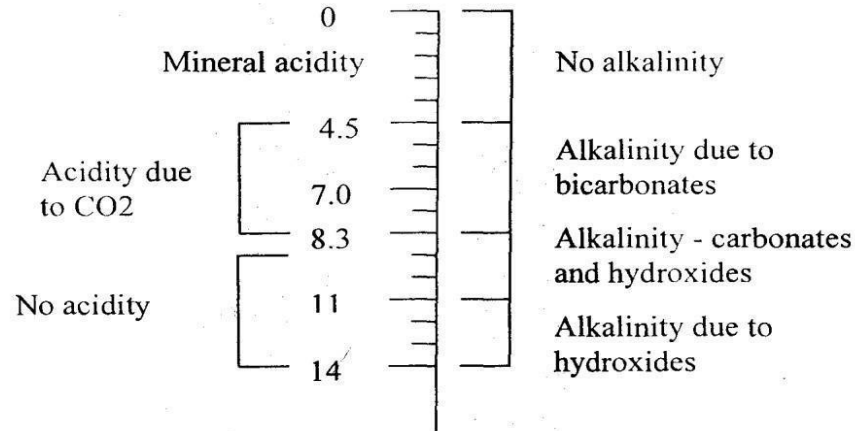
Turbidity

It is a measure of degree of opaqueness of water and interference presented by suspended matter to the passage of light. The turbidity is due to clay, silt, finely divided organic matter and microscopic organisms. Turbidity tests are important from aesthetic consideration and from the point of economics of treatment. The most important health significance of turbidity is that may, harbour pathogenic organisms.

pH

Determinations of pH, alkalinity and its forms, along with acidity are of interest in coagulation, softening and corrosion control

pH scale



Residue or solid matter

The test for residue is of very great importance in sewage treatment processes to indicate the physical state of the principal constituents. The ratio of the weight of suspended solids to turbidity often referred as coefficient of fineness. The solids present in dissolved form are related to the electrical conductivity. The fixed solids indicate the mineral level while volatile solids are related to organic matter.

Chloride

Concentration of chlorides in municipal sewage is often significantly (15-50 mg/L) higher than those in its water supply. For this reason, a change in its concentration

may be indicative of sewage pollution, in waters of low chloride concentration.

Chlorides

occur in an all natural waters in widely varying amounts. Mountain streams are normally low in chloride values. Chloride gain access to water either because of excellent solvent properties or through human excreta or industrial pollutants. Chlorides were for several years used as an indicator of pollution by municipal wastes in rivers, streams, wells and lakes.

Dissolved Oxygen

In raw water and domestic wastes, dissolved oxygen is a factor which determines whether the biological processes undergoing a change are aerobic or anaerobic. It is very desirable that aerobic conditions are maintained. It is a Single test which will immediately indicate the sanitary status of a stream. Low values of dissolved oxygen adversely affect the portability of water and may cause fish kill.

Organic matter

The tests of organic matter indicate type and extent of pollution, which has its origin in plant or animal matter. Tests are mostly restricted to the study of nitrogen in various forms and oxygen requirements in biodegradation of putrescible carbonaceous organic matter (BOD). A measure of the demand is also indicated in terms of demand through strong chemical oxidants (COD)

BOD.

The BOD is the amount of oxygen required by bacteria while stabilizing decomposable organic matter under aerobic conditions. Polluted water does not contain sufficient oxygen in solution to maintain aerobic condition during decomposition. The quantity of oxygen required for complete stabilization is taken as a measure of its organic content.

CODP

The COD test is based on the concept that a large majority of organic compounds can be completely oxidized by the action of strong oxidizing agents in acidic medium. The quantity of oxygen required is proportional to organic matter, regardless of the biological assimilability of the substance.

Nitrogen

Nitrogen is estimated as organic nitrogen, ammonical nitrogen, nitrite nitrogen and nitrate nitrogen throw light on the pollutional history of the carrying water.

Bacteriological tests

The routine bacteriological tests are aimed at enumerating the members of coliform group, which are considered indicators of pollution. The natural habitat of these bacteria is the intestinal tract of man and other warm blooded animals. They are present wherever the pathogens are present and by their absence exclude the probability of the presence of pathogens. They share the fate of the most significant pathogenic enteric bacteria outside the human and animal body both in the rate of death and in the rate of removal when water is purified.

Another test of bacteria is aimed at detecting chemo-synthetic heterotrophic heterogeneous group developing under conditions of cultivation and is referred as

Total Plate Count. This test is not differential and indicates a total picture of bacteria associated with organic matter.

Biological Examination

The biological examination (microscopic) provides useful information for the control of water quality and treatment. It serves for one or several of the following purposes:

- i) To explain the cause of color or an odor in water
- ii) To aid in the interpretation of various chemical analysis reports
- iii) Permitting identification of specific water when it is mixed with another
- iv) To explain clogging of pipes/screens/filters
- v) Rapidly detect organic pollution and contamination with toxic substances
- vi) To indicate the progress of self purification streams.

Observation

Source of sample :

Date of collection:

Time :

Temperature:

Tabulation

Sample	Calibration with	Temperature	pH value	Remark

Result

Thus the sampling and preservation methods in water and waste water characterization and to learn the significance of characterisation of water and waste water are studies.

Ex No :2

Determination of pH

Date :

Aim:

To determine the pH value of the given sample by electrometric method.

Apparatus Required

pH meter with combined electrode, beakers

Chemicals Required

Buffer tablet of pH values 4 and 9.2

Reagents preparation

Buffer solution of pH value 4

Buffer tablet of pH value 4 is dissolved in 100 ml of distilled water. This solution should preferably be stored in a plastic bottle in cool place.

Buffer solution of pH value 9.2

Buffer tablet of pH value 9.2 is dissolved in 100 ml of distilled water. This solution should preferably be stored in a plastic bottle in cool place.

Procedure:

Electrometric method:

1. Wash the combined electrode of pH meter with distilled water and clean the same with distilled water.
2. Dip the combined electrode in the buffer solution of pH value 4.
3. Adjust the temperature by the adjustment knob to an ambient (room) temperature.
4. If the instrument shows the reading as 4 then it is in order if not, adjust the reading to 4.0 by calibration adjustment knob.
5. Wash the electrode of pH meter with distilled water and clean the same with distilled water and dip it to the buffer solution of pH value 9.2.
6. Note the reading if the instrument shows the reading as 9.2 then it is in order otherwise use the calibration adjustment knob and bring the reading to 9.2.
7. Repeat the above procedure until the meter shows reading as 4 when electrode is dip in buffer solution of pH 4 and shows reading as 9.2 when electrode is dip in buffer solution of pH value 9.2.
8. Now the instrument is calibrated.

9. After cleaning the electrode dip in the sample for which pH value is to be found out.
10. Directly record the reading from the meter without doing any adjustments.

Environmental significance

pH (6.5 to 8.5) has no direct effect on health however a lower value below 4 will produce sour taste and higher value above 8.5 a bitter taste. Higher values of pH have scale formation in water heating operators and also reduce the germicidal potential of chlorine. High pH induces the formation of trihalomethanes which are causing cancer in human beings.

pH below 6.5 starts corrosion in pipes, thereby releasing toxic metals such as zinc, lead, cadmium & copper etc., According to BIS water for domestic consumption should have pH between 6.5 to 8.5

Application of pH data in environmental engineering practice

1. Determination of pH is one of the important objective in biological treatment, if the pH goes below 5 due to excess accumulation of acids, the process is severely affected. Shifting of pH beyond 5 to 10 upsets the aerobic treatment of the waste waters. In these circumstances, the pH can be adjusted by addition of suitable acid or alkali to optimize the treatment of waste water.
2. Its range is of immense value for any chemical reaction. A chemical value shall be highly effective at particular pH. Chemical coagulation; disinfection, water softening and corrosion control are governed by pH adjustment.
3. Dewatering of sludges, oxidation of cyanides and reduction of hexa valent chromium in to trivalent chromium also need a favorable range 4. It is used in the calculation of carbonate, bicarbonate, CO₂ calculation, stability index and acid- base equilibrium.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Jar No	Amount of coagulant added (g)	Floc formation (ml)

Result:

The pH value of the given sample by electrometric method is _____

Ex No: 3

Optimum Coagulant Dosage by Jar Test

Date :

Aim:

To determine the optimum dosage of coagulant required for a given sample of waste water.

Apparatus Required:

1. Laboratory flocculator with stirring paddles
2. Glass Jars
3. Analytical balance

Reagents Required: Alum

Theory:

The amount of coagulant required for coagulation depends on the turbidity of the waste water. The use of optimum amount of coagulant is indicated by the formation of the large feathery flakes. This can be approximately determined in the laboratory by Jar test. The test involves rapid mixing to disperse the chemicals (coagulants) in the sample and slow mixing for the floc formation.

PROCEDURE:

1. Fill 1 litre waste water sample in each of the six jars
2. Attach the sample jars to the stirring device by lifting the paddles in the right upward direction.
3. Add coagulant (Alum) in progressive dosages into the series of the six sample jars.
4. The coagulant dosage can be selected randomly depending on the characteristics of waste water.
5. Mix the sample rapidly for about 10 mm with mechanically operated paddles at 180rpm followed by gentle stirring about 10 mm. at 30—40 rpm
6. Remove the jars from the stirring device after stirring is completed
7. Let the sample in the jars stand for 30 mm. for settling of floc.
8. The dose of coagulant versus floc formation is plotted as graph.
9. The dose of coagulant which gives the best floc is the optimum dose of coagulants.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	quantity of water sample used in ml	Test on water sample ml (Burette Reading)			Test on water sample ml (Burette Reading)			Chlorine residuals Mg/L
		Initial	Final	Na ₂ S ₂ O ₃ Used (x ml)	Initial	Final	Na ₂ S ₂ O ₃ Used (y ml)	

Calculation

Residual Chlorine in original water sample

$$= (x - y) \times 1000 \times 0.8895$$

$$= \frac{\text{ml of water sample}}{\text{Mg/L}}$$

Result:
The optimum coagulant dosage _____ mg/L

Ex No : 4

DETERMINATION OF RESIDUAL CHLORINE

Date

Aim

To determine the residual chlorine for the given water sample

Apparatus Required

- Burette with stand, tiles
- Pipette
- Conical flask
- Beaker
- Glass funnel
- Measuring jar

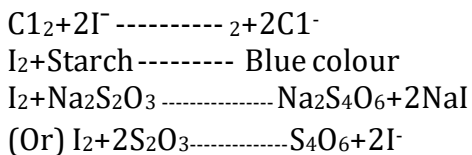
Principle

Chlorine combines with water to form Hypochlorous and Hydrochloric acids
 $\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{H}^+ + \text{Cl}^-$

In water chlorine, hypochlorous acid and hypo chlorite ions are referred as free chlorine residuals and the chloramines are called combined chlorine residuals.

The chlorine demand of water is the amount of chlorine that must be supplied to leave a desired free combined or total residual after a specified contact period.

The starch iodide test is an age old method for testing the total chlorine residual in a given water sample and is still being used, depending upon the oxidizing power of free and combined chlorine to convert iodide ion to free iodide. This free iodine liberates iodine ions when titrated with sodium thiosulphate as shown below;



Reagents Required

Chlorine water 1 gmL /
Acetic Acid
Standard N/40 Sodium thiosulphate solution
Starch indicator

Reagents Preparation:

Starch Indicator:

Weigh 1 g of starch and make it into a paste with 10 ml of hot water and dilute it to 100 ml.

Standard N/40 Sodium Thiosulphate solution:

Dissolve 1.575 gm of $\text{Na}_2\text{S}_2\text{O}_3$ in distilled water and make up to 1 litre. Procedure

1. Take 25 ml of given water sample in a conical flask.
2. Add a small crystal of KI and distilled water to the above flask containing water sample, to make 100 ml.
3. Add about 0.5 ml conc. HCl or about 100 ml acetic acid to act as buffer to reduce the pH to a low value between 3.5 to 4.2 to avoid conversion of Cl_2 into HOCl and OCl^- .
4. Titrate the above yellow coloured iodine solution against standard Sodium thio sulphate till yellow becomes light or faintly yellow.
5. Add 1 ml of soluble starch solution (end point indicator) to change the colour from light yellow to blue in the conical flask.
6. Continue the titration with standard sodium thiosulphate till the blue colour just disappears.
7. Note down the total amount of titrant used in the entire titration. Let it be x ml.
8. Repeat the titration with distilled water as sample and determine the amount of sodium thiosulphate. Let it be y ml.

Environmental significance

The residual chlorine is the measure of chlorine left in water after the required contact period, which will ensure complete killing of bacteria and oxidation of the — organic matter. Usually a free chlorine residue of 0.2 to 0.3 mg/L after a contact period of 10-20 minutes is considered to be sufficient and satisfactory to take care of the future contamination of water to be supplied through the distribution system.

OBSERVATION :

Source of sample :

Date of collection :

Time :

Temperature :

Tabulation

S.No.	Volume of water sample (ml)	Burette Reading (ml)		EDTA Consumed (ml)	Hardness (mg/L)
		Initial	Final		

Calculation

$$\text{Hardness in mg/1 of CaCO}_3 = \frac{\text{ml of EDTA used}}{\text{ml of water sample}} \times 1000$$

Result

Residual Chlorine residuals for given water sample by Starch Iodide Test is = _____mg/L.

Ex. No: 5

DETERMINATION OF HARDNESS

Date :

Aim

To determine the total hardness present in the given sample

Apparatus Required

Burette, Pipette, Conical flask, measuring jar

Chemicals Required

Eriochrome Black-T (EBT) indicator, Ammonium chloride, Ammonium solution, EDTA

Reagents Preparation

EBT indicator

Dissolve 0.2 g of pure solid in 15 ml of distilled water

Standard EDTA Titrant

Take 0.5 g of EDTA. Heat it to 80°C for half an hour and cool it. Take 0.37 g of the dissolve and add to get distilled water to make 100ml.

1 ml of exactly 0.02 N EDTA 1mg of CaCO_3

Ammonia Buffer solution

Dissolve 0.7 g of ammonium chloride (NH_4Cl) in 5.7ml concentrate ammonia solution and dilute to 100 ml.

Procedure

1. Take 20 ml of the sample in a conical flask.
2. Add 2ml of ammonia buffer to the flask.
3. Add 5 — 6 drops of EBT indicator to the flask wine red colour will be developed.
4. Titrate it with standard EDTA solution which is filled in the burette till the colour changes from wine red to blue.
5. Repeat steps I to 4 for different samples with varying hardness and also for distilled water (blank)
6. For determination of non-carbonate hardness the sample is to be boiled for 30 minutes. The procedure is the same as above.

Sanitary Significance

Hard water has adverse action with soap since it allows less formation of lather. If hard water is used in boilers, scaling problem occurs leading to the bursting of boilers, It makes food tasteless. It affects the working of dyeing process. It is also precipitate protein of meat and make tasteless.

Application of Hardness data in Environmental Engineering Practice

1. Hardness of water is important in determining the suitability of water for domestic and industrial uses.
2. The relative amounts of calcium and magnesium hardness, carbonate and non-carbonate hardness present in water are the factors while determining the most economical type of softening process.
3. Determination of hardness serve as a basis for routine control of softening process.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

Silver nitrate Vs Sodium Chloride

S.No.	Volume of water sample (ml)	Burette Reading (ml)		AgNO ₃ consumed (ml)
		Initial	Final	

Concordant Value = ml

Silver Nitrate Vs sample

S.No.	Volume of water sample (ml)	Burette Reading (ml)		AgNO ₃ consumed (ml)	Chlorides (mg/L)
		Initial	Final		

Concordant Value = ml

Calculation

$$\text{Normality of silver Nitrate } N = \frac{\text{Volume Normality of NaCl}}{\text{Vol. of AgNO}_3}$$

$$\text{Chloride mg/L} = \frac{\text{Volume of AgNO}_3 \times 35.45 \times N \times 1000}{\text{Vol. of sample taken}}$$

Result:

Total Hardness in mg of CaCO₃ = ----- mg/L

Ex No : 6

Determination of chloride

Date :

Aim

To determine the amount of chloride present in the given sample

Apparatus required

Burette with stand, pipette, conical flask measuring jar etc.,

Chemicals Required

Sodium Chloride, Silver nitrate, Potassium Chromate

Reagents preparation

Silver Nitrate Solution

Dissolve 1.2g of silver nitrate in distilled water and make up to 250 ml.

Sodium chloride Solution (0.028N)

Dissolve 0.1648g of sodium chloride in distilled water and make up to 100ml.

Potassium Chromate Solution (K₂CrO₄)

Dissolve 1 gm of potassium chromate in 20ml of distilled water.

Procedure

Standardization of Silver Nitrate Solution

1. Pipette 20 ml of sodium chloride solution into the conical flask.
2. Add one or two drops of potassium chromate solution.
3. Titrate against Silver Nitrate solution until the appearance of reddish brown colour
4. Repeat the titration for concordant values.

Silver Nitrate Vs Sample -

1. Pipette 20 ml of sample into the conical flask.
2. Add one or two drops of potassium chromate solution
3. Titrate against silver Nitrate solution until the appearance of reddish brown colour.
4. Repeat the titration for concordant values.

Environmental Significance of Chlorides

Chloride associated with sodium exerts salty taste, when its concentration is more than 250 mg/l. There is no known evidence that chloride-constitute any human health hazard. For this reason, chlorides are generally limited to 250 mg/L in supplies intended for public use. In many areas of world where water supplies are scarce, sources containing as much as 2000mg/L are used for domestic purposes without the development of adverse effect once the human system becomes adapted to the water.

It can also corrode concrete by extracting calcium in the form of calcide. Magnesium chloride in water generates hydrochloric acid after heating which is also highly corrosive and create problems in boilers.

Application of chlorides data in environmental engineering practice

1. Chlorides determination in natural waters is useful in the selection of water supplies for human use.
2. Chlorides determination is used to determine the type of desalting operators to be used.
3. The chloride determination is used to control pumping of ground water from locations where intrusion of sea water is a problem.
4. Chlorides interfere in the determination of COD a correction must be made on the basis of the amount of chloride present. -

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Sample details	Turbidity (NTU)

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

Where

A= NTU of diluted sample
B=Volume of dilution water
C=Sample volume taken for dilution, ml.

Result:

Amount of chloride present in the given sample= _____ mg/L

Ex No : 7

TURBIDITY

Date :

Aim

To find out the turbidity of the given sample.

Principle:

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

Apparatus Required:

Nephelometers turbidimeter, Sample tubes.

Reagents Preparation

1. Dissolve 1.0gm Hydrazine sulphate and dilute to 100ml
2. Dissolve 10gm Hexa methylene Tetra min& and dilute in 100ml
3. 5ml of each of the above solution (1 and 2) in a 100ml volumetric flask and allow to stand for 24 hrs at $25\pm 3^{\circ}\text{C}$ and dilute to 1000ml. This solution has a turbidity of 40NTU.

Procedure:

1. The Nephelometer turbidimeter is switched on and waited for few minutes till it warms up.
2. The instrument is set up with a 40NTU standard suspension
3. The sample is thoroughly shaken and kept it for sometimes so the air bubbles are eliminated
4. The sample is taken in Nephelometer sample tube and the sample is put in Sample chamber and the reading is noted directly.
5. The sample is diluted with turbidity free water and again the turbidity is read.

Environmental Significance

Turbidity is objectionable because of

- a. Aesthetic considerations and
- b. Engineering considerations

When turbid water in a small, transport container. such as drinking glass is held up to the light, an aesthetically displeasing opaqueness or 'milky' coloration is apparent

The colloidal material which exerts turbidity provides adsorption sites for chemicals that may be harmful or cause undesirable tastes and odours & for biological organisms that may be harmful. Disinfections of turbid water is difficult because of the adsorptive characteristics of some colloids and because the solids may partially shield organisms from disinfectant.

In natural water bodies, turbidity may impart a brown or other colour to water and may interfere with light penetration and photosynthetic reaction in streams and lakes.

Turbidity increases the load on slow sand filters. The filter may go out of operation, if excess turbidity exists.

Application of Turbidity Data in Environmental Engineering Practice:

Turbidity measurements of particular importance in the field of water supply. They have limited use in the field of domestic and Industrial waste treatment.

1. Knowledge of the turbidity variation in raw water supplies along with other information is useful to determine whether a supply requires Special treatment by chemical coagulation and filtration before it may be used for a public water supply.
2. Turbidity measurements are used to determine the effectiveness of the treatment produced with different chemicals and the dosages needed.
3. Turbidity measurements help to gauge the amount of chemicals needed from day-today in the operation of water treatment works.
4. Measurement of turbidity is settled water prior to filtration is useful in controlling chemical dosages so as to prevent excessive loading or rapid sand filters.
5. Turbidity measurements of the filtered water are needed to check o faculty filter operation.
6. Turbidity measurements are useful to determine the optimum dosage of coagulants to treat the domestic and Industrial wastes.
7. Turbidity determination is used to evaluate the performance of waste treatment plants.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Burette Reading (ml)		Vol Na ₂ S ₂ O ₃ consumed X (ml)	% of chlorine in powder sample = <u>X*0.71</u> M
	Initial	Final		

Calculation:

**Chlorine in mg/L in chlorine solution = ml of Na₂S₂O₃ consumed i.e
X*35.5*1000*.025**

ml of chlorine solution

$$\begin{aligned} \text{\% age chlorine in sample} &= \frac{\text{mg/1 of chlorine}}{\text{Mg/1 of powder}} \times 100 \\ &= \frac{\text{X} \times 35.5 \times 100}{5000 \text{ M}} \\ &= \text{X} \times 0.71 \text{ \%} \\ &\text{gm of sample used} \end{aligned}$$

Result:

**The turbidity of Tap water =
Synthetic sample =**

Ex No : 8

**DETERMINATION OF AVAILABLE
CHLORINE IN BLEACHING POWDER**

Date :

Aim:

To determine the available chlorine percentage in a given sample of bleaching powder

Theory:

In order to work out the disinfecting power of bleaching powder and its requirement for treating given water having a particular chlorine demand, we have to work out the chlorine content in the given bleaching powder.

Reagents Preparation:

Starch Indicator:

Weigh 1 g of starch and make it into a paste with 10 ml of hot water and dilute it to 100 ml.

Standard N/40 Sodium thiosulphate solution:

Dissolve 1.575 gm of $\text{Na}_2\text{S}_2\text{O}_3$ in distilled water and make up to 1 litre.

Procedure:

1. Take 0.7 gm of bleaching powder in to a beaker.
2. Thoroughly mix the powder with distilled water in the beaker and pour the solution with several rinsing into a 200ml volumetric flask.
3. Fill the 200ml flask with distilled water up to the mark, as to make the chlorine solution equal to 200ml. Mix thoroughly.
4. Dissolve about 2 gm of potassium iodide and 2ml of glacial acetic acid in 2ml of distilled water in a conical flask.
5. Pipette out 25ml of the chlorine solution from the volumetric flask and add it into the above flask containing iodide acetic acid mix.
6. Add a few drops of starch indicator to the conical flask to develop blue colour.
7. Titrate this blue solution in the conical flask against the sodium thiosulphate solution, till the blue colour in the flask just disappears.
8. Note down the ml of sodium thiosulphate solution used in the above titration. It may be averaged out by performing the above test twice or thrice. Let this value be Xml.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Vol. of given sample ml	Weight of empty crucible W₁gm	Weight after one heating W₂gm	Weight after two heating W₃gm

$$\frac{W_2 - W_1}{\text{Volume of sample}} = \text{Total solids in mg/l}$$

Result:

The percentage chlorine content of the given bleaching powder works out to be _____

Ex No : 9

TOTAL, FIXED AND VOLATILE SOLIDS

Date

Aim:

To determine the amount of total, fixed and volatile solids present in the given sample.

Apparatus Required:

Crucible, Chemical balance, hot air oven, muffle furnace, desicator

Procedure:

1. Take the empty crucible. Clean it thoroughly and make it perfectly dry. Take the weight of empty crucible.
2. Add to the crucible 20ml of liquid sample.
3. Heat the crucible in water bath at 100°C till the entire liquid in a crucible evaporates and dry residue remains at the bottom then place the crucible in oven at 103°C for 1 hour.
4. Take the weight of the crucible with residue after cooling it in a desicator for 20 minutes. Let us weight be W_2 gm.
5. Take the sample crucible and keep it in a muffle furnace at a temperature of 650°C for 30min
6. The volatile and organic matter in the solids evaporated and the crucible contains only fixed solids.
7. Cool the crucible in a desicator and weight it with the fixed solids residue. Let the weight be W_3 gm

Environmental Engineering Significance:

The water which contains of high volatile solids is not suitable for drinking purposes. The result of high volatile solids indicates that the water may have been pollutes by domestic waste or other organic waste. In general, ground water is free from volatile solids unless they have been polluted by waste seepages. But, well water may have high volatile solids due to lack of proper protection around well to prevent seepage of used water. Surface water may also have high volatile solids due to disposal of domestic and other wastes.

Application in Environmental Engineering Practice:

Volatile solids test is normally applied to sludges.

It is indispensable in the design operation of sludge desicator, vacuum filter and infiltration plants.

Before the development of C.O.D. test it is used to find the strength of industrial and domestic waste water.

It is helpful in accessing the amount biologic ally inert organic matter, such as lignin in the case of wood pulping waste liuor.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Volume of given sample after filtration ml	Weight of empty crucible W_1	Weight after one heating W_2 gm

Dissolved solids in mg/1 =
$$\frac{W_2 - W_1}{\text{Vol of sample}} \times 10^6$$

Suspended solids = Total solids - Dissolved solids

Result:

Total Solids = _____
Fixed solids = _____
Volatile solids = _____

Ex No : 10

Suspended and Dissolved Solids

Date

Aim:

To determine the amount of dissolved solids present in the given sample.

Apparatus Required:

Crucible, oven, desiccator, chemical balance.

Procedure:

1. Take a known quantity of liquid sample in a crucible of known weight.
2. The sample is filtered through walt man paper number 44. The dissolved solids go in solution through the filter paper.
3. Take a known quantity of filtered solution in a crucible of known weight and dry it to a temperature of 103°C to 105°C.
4. Cool the crucible in a desiccator and weigh it let the weight be W_2 gms. /

Environmental Significance:

Water with high dissolved solids generally is of inferior palatability and makes it use an unfavorable physiological reaction in a transit consumer.

Suspended Solids containing much organic matter make as purification and consequently this may be dividing of dissolved oxygen loading to destruction of plant and human life.

Application:

Dissolved solids determination gives an idea about the formation of scales cause of foaming in boilers, acceleration of corrosion and interference with the colour and taste of many finishes products.

The suspended solid determination is particularly useful in the analysis of sewage and other waste water. It is used to evaluate the strength of waste water and to determine the efficiency of treatment units.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Sample details	Volume of given sample ml	Total settlable solids ml/1/hr

Result:

Solids present in the given sample
Dissolved solids = _____
Suspended solids= _____

Ex No: 11

TOTAL SETTLABLE SOLIDS

Date

Aim:

To find out the total settleable solids of the given sample.

Apparatus Required:

Imhoff cone, holding device

Procedure:

1. The imhoff cone is gently filled with the thoroughly well mixed sample usually 1 litre and allowed it to settle.
2. After 45 minutes, the cone is rotated between hands to ensure that all solids adhering to the sides are loosened.
3. The solids are allowed to settle for 15 mm more to make up for a total period of 1 hour.
4. The volume of the sludge which has settled in the open is noted.
5. The results are expressed in ml settleable solids per litre of sample per hour.

Precautions:

1. The imhoff cones must be cleaned with a strong soap and hot water using a brush.
2. The cone is wetter before use, which helps in preventing adherence of the solids to the sides.
3. The method is subjected to considerable inaccuracy if the solids contain large fragments.
4. The determination of total settleable solids should be carried out soon after sampling in order to avoid errors through flocculation.

Application:

1. The settleable solids determination is used extensively in the analysis of industrial waste to determine the need for and design of plain settling tank in plants employing biological treatment process.
2. It is also widely used in waste water treatment plant operation to determine the efficiency of sedimentation tanks.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

Sodium thiosulphate Vs given sample

S.No.	Volume of given sample ml	Burette Reading (ml)		Indicator	Endpoint
		Initial	Final		

Calculation :

100 ml of N thiosulphate solution = 8 gms of oxygen

Dissolved Oxygen in mg/L

$$= \frac{V_2 \times N \times 8 \times 1000}{V_1}$$

V₁ = Volume of water sample in ml.

V₂ = Volume of Sodium thiosulphate in ml.

N = Normality of sodium thiosulphate

Result:

The total settleable solids is= _____ ml/l/hr

Ex No: 12

DETERMINATION OF DISSOLVED OXYGEN

Date:

Aim:

To determine the amount of Dissolved Oxygen present in the given sample.

Apparatus Required:

Burette with stand ,pipette,conical flask,measuring jar

Chemicals Required:

Sodium Hydroxide, Manganous Sulphate, Potassium iodide, Sodium Thiosulphate, Conc.H₂SO₄, Starch

Reagent Preparation:

1. Manganous Sulphate:

12 gms of Manganous Sulphate is dissolved in 25m¹ of distilled water.

2. Alkaline —Iodide Solution

9 gms of Sodium Hydroxide and 2.5gms of Potassium iodide are dissolved in 25m¹ of distilled water.

3. Sodium thiosulphate Solution (0.01N)

2.48gms of Sodium thiosulphate is dissolved in 1 litre of water.

4. Starch Solution

Take 1 gm of starch. Prepare paste with distilled water. Make 100 ml with water and boil by stirring and cool it.

5. Pipette Solution:

2m¹ of Manganous Sulphate solution and 2ml of alkaline Iodide Solution is added to 250m¹ of the sample taken in a reagent bottle. The bottle is stoppered and shaken thoroughly when the precipitate formed is settled, 2ml of Cone. HCL or Conc. H₂SO₄ is added and shaken thoroughly until the precipitate gets dissolved completely.

Procedure:

1. Take 50ml of clear pipette solution in a conical flask

2. Add to it one or two drops of starch indicator until the colour becomes blue.

3. Titrate against Standard Sodium Thiosulphate solution until the disappearance of colour.

4. Repeat the titration for concordant values.

Sanitary Significance:

In liquid wastes Dissolved Oxygen is the most important factor in determining whether aerobic or anaerobic organisms carryout biological changes. If sufficient D.O is available aerobic organisms oxidize the wastes to stable products. If D.O is deficient anaerobic bacteria take part in the conversion and reduce the waste often to obnoxious and nuisance conditions are usually resulted.

Application in Environmental Data:

1. It is one of the most important tests often used in most instances involving stream pollution control.
2. For the survival of aquatic life maintenance of D.O level is a must.
3. Determination of D.O serve as the basis of B.O.D test and thus they are the foundation of the most important determination used to evaluate pollution strength of sewage and industrial waste.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

S.No.	Weight of empty crucible(g) A	Weight of crucible after heated in oven(g) B

Calculation:

Volume of sample taken = 50ml
Weight of empty silica crucible = Ag
Weight of crucible+precipitate=Bg
233g of BaSO4 contains 96g of SO4
(B-A) g of BaSO4 will contain = $\frac{96 \times (B-A)}{233}$
In 10₆ ml = $\frac{96 \times (B-A) \times 10^6}{233 \times 50}$ ppm
In terms of me/litre = ppm/48.

Result:

Amount of Dissolved Oxygen present in a given sample is _____

Ex No: 13

ESTIMATION OF SULPHATES

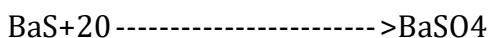
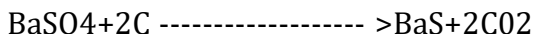
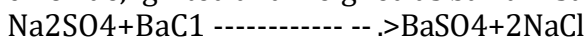
Date

Aim:

To determine the amount of sulphate present in the given sample by gravimetric method.

Principle:

The sulphate in water is precipitated as Barium Sulphate by the addition of Barium Chloride in hydrochloric acid medium. The precipitate is filtered, washed free of chloride, ignited and weighed as barium sulphate.



Apparatus Required:

Crucible, Oven, weighing balance, pipette, beaker, water bath, desiccator.

Reagents Required:

Dilute HCl

Solid Ammonium Chloride

10% Barium Chloride solution

Reagents Preparation:

Barium Chloride Solution:

Dissolve 10g of Barium Chloride in 100 ml of distilled water.

Procedure:

1. Pipette out 50ml of sample into a clean 250ml beaker.
2. Add 10ml of dilute HCl & 1gm of solid ammonium chloride.
3. Heat to boiling & add 10ml of 10% Barium Chloride solution drop by drop with constant stirring. Continue boiling for another 2 to 3 minutes.
4. Allow the precipitate to settle and test for completion of precipitation by adding a small amount of Barium Chloride solution through the sides of the beaker.
5. If any turbidity is noticed add sufficient quantity of barium chloride to precipitate all the sulphate.
6. Transfer the contents to a sand bath & digest for half an hour to promote granulation of the precipitate.
7. Filter through Whatman no 42 filter paper and wash with boiling water till the filtrate runs free of chloride.
8. Transfer the filter paper along with the precipitate to a weighed silica crucible and dry it in an air oven.
9. Ignite over a low flame initially, taking care to ash the filter paper completely, then ignite strongly over a rose head flame to constant weight.
10. From the weight of Barium Sulphate obtained calculate the Sulphate content of the sample.

Environmental Significance:

Sulphates in natural waters in concentrations ranging from a few to thousand mg/L. Excess Na_2SO_4 and MgSO_4 should not be present in drinking waters as they cause Cathartic action. Higher Concentration of Sodium Sulphate in water can cause malfunctioning of the alimentary canal. So the recommended upper limit is 250mg/l in water intended for human consumption. In anaerobic decomposition of waste waters, Sulphates are reduced to hydrogen Sulphide causing obnoxious odours and promote corrosion of sewers. Sulphates are reduced to sulphide in sludge digesters and may upset the biological process, if the sulphide concentration exceeds 200mg/L.

Application of Sulphate Data in Environmental Engineering Practice:

1. The sulphate content of natural waters is an important consideration in determining their suitability for public and industrial water supplies.
2. The amount of sulphate in waste water is a factor of concern in determining the magnitude of problems that can arise from reduction of sulphates to hydrogen sulphide.
3. A knowledge of the sulphate content of the sludge or waste fed to digestion units provides a means of estimating the hydrogen sulphide content of the gas produced. From this information, the design engineer can determine whether scrubbing facilities will be needed to remove hydrogen sulphides and size of the units required.

Result:

The amount of Sulphate present in the given sample _____ mg/L.

Ex No: 14

DETERMINATION OF FLUORIDES

Date :

Aim:

To determine the fluorides present in the give sample.

Apparatus Required:

Burette with stand, Pipette, Conical flask, measuring flask.

Chemical required:

Oxalate, concentrated hydrochloric acid, phenolphthalein indicator, sodium hydroxide.

Reagents Preparation:

Oxalate Solution:

Dissolve 630mg of oxalate in distilled water and make up to 100ml.

Phenolphthalein indicator:

Add 1g of phenolphthalein in 200 ml distilled water and dissolve it. Add 0.02N Sodium hydroxide solution drop wise until a faint pink colour appears.

Sodium hydroxide solution:

Dissolve 4g of sodium hydroxide in distilled water and make up to 100ml.

Procedure:

Titration — I NaoH Vs Oxalic acid

1. Pipette 20ml of oxalic acid solution into the conical flask.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Titration —II NaoH Vs Sample:

1. Take 10ml of sample in the conical flask and add 1 ml of concentrated hydrochloric acid.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

OBSERVATION :
Source of sample :
Date of collection :
Time :
Temperature :

Tabulation

Titration - I NaoH Vs Oxalic acid

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Oxalic acid					Appearance of pale pink colour

Titration - II NaoH Vs Sample

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Tap Water					Appearance of pale pink colour

Titration - III NaoH Vs HCL

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Conc .HCL					Appearance of pale pink colour

Calculation:

Titration I

Volume of oxalic acid × Normality of oxalic acid

Normality of NaOH X = _____

Volume of NaOH

Titration II

Volume of NaOH × Normality of NaOH

Normality of Sample Y =

Volume of sample

Titration II-III × X × Y

Amount of Fluorides =

Volume of Sample

Titration — III NaOH Vs HCl:

1. Take 19ml of distilled water in the conical flask and add 1ml of concentrated hydrochloric acid.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Environmental Significance Fluorine in Human health

Presence of large amount of fluoride is associated with dental and skeletal fluorosis (>1.5 mg/i) and in adequate amounts with dental caries (<1 mg/i)

Dental Fluorosis

In young children the disease affects only on the teeth. This is known as dental fluorosis. The teeth lose their shiny appearance and chalk-white patches develop on them. This is known as mottled enamel and is an early sign of dental fluorosis. The white patches later become yellow and turn brown or black.

Skeletal Fluorosis

In aged people the disease affects the bones, tendons and ligaments. This is known as skeletal fluorosis. This is followed by pain and stiff of the back and later the joints of both limbs and limitation of neck movements.

Genu Valgum

It was observed that this syndrome was most prevalent among people whose staple diet was sorghum. Further studies have shown that diets based upon jower promote a higher retention of ingested fluoride than their based on rice.

Application of fluoride data in Environmental Engineering Practice:

1. Fluoride of water is an important in determining the suitability of water from domestic and industrial uses.
2. The size and design of Deflouridation units depends upon the level of fluorides present in the water.

OBSERVATION :

Source of sample :

Date of collection :

Time :

Temperature :

Tabulation

Titration - I NaoH Vs Oxalic acid

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Oxalic acid					Appearance of pale pink colour

Concordant Value = _____ ml

Titration - II NaoH Vs Sample

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Ammonia solution					Appearance of pale pink colour

Concordant Value = _____ ml

Titration - III NaoH Vs HCL

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Sewage					Appearance of pale pink colour

Concordant Value = _____ ml

-

Result:

Amount of fluorides present in the given sample (Tap water)
_____mg/L.

Ex.No:15

DETERMINATION OF AMMONIACAL NITROGEN

Date:

Aim:

To determine the ammoniacal nitrogen present in the given sample.

Apparatus Required:

Burette with stand, Pipette, Conical flask, measuring flask.

Chemical required:

Oxalate, concentrated hydrochloric acid, phenolphthalein indicator, sodium hydroxide, ammonia solution.

Reagents Preparation:

Oxalate Solution:

Dissolve 6.30g of oxalate in distilled water and make up to 100ml.

Phenolphthalein indicator:

Add 1g of phenolphthalein in 200 ml distilled water and dissolve it. Add 0.02N Sodium hydroxide solution drop wise until a faint pink colour appears.

Sodium hydroxide solution:

Dissolve 4g of sodium hydroxide in distilled water and make up to 100ml.

Standard Hydrochloric acid:

Dissolve 2 ml of HCl in distilled water and make up to 100 ml.

Procedure:

Titration — I NaOH Vs Oxalic acid

1. Pipette 20ml of oxalic acid solution into the conical flask
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Titration —II NaOH Vs Ammonia

1. Take 17ml of distilled water in the conical flask and add 1ml of ammonia solution and 2 ml of hydrochloric acid.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Calculation:

Titration I

Volume of oxalic acid × Normality of oxalic acid (0.1)

Normality of NaOH = _____

Volume of NaOH

Titration II

Volume of NaOH × Normality of NaOH × 0.0091

Normality of Sample X =

Volume of sample

$$\frac{\text{Amount of Ammoniacal Nitrogen in mg/L} \times \text{X} \times \text{Vol of NaOH}}{\text{Volume of Sample}} \times 1000$$

Titration — III NaOH Vs sample

1. Take 17ml of distilled water in the conical flask and add 1ml of sample and 2 ml of hydrochloric acid.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Environmental Significance

1. Excess of ammonia in the form of nitrogen leads to Eutrophication in lakes.
2. Consumption of Nitrogen greater than 2mg/l in drinking water may lead to methemoglobinemia in children.

APPLICATION

1. Determination of ammoniacal nitrogen used for standardizing the drinking water supply.
2. The data is used in the treatment of waste water before it is subjected to water courses.
3. It is also used to determine the extent of eutrophication and possible methods of removal of Nitrogen.

OBSERVATION :

Source of sample :

Date of collection :

Time :

Temperature :

Tabulation

Titration - I NaoH Vs Oxalic acid

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		

Concordant Value = _____ ml

Calculation:

$$\text{COD mg/L} = \frac{(A-B) C \times 8 \times 1000}{\text{ml.of sample used}}$$

Where

A = ml of ferrous ammonium sulphate used for balnk

B = ml of ferrous ammonium sulphate used for sample

C = Normality of ferrous ammonium sulphate solution

Result:

Amount of Ammoniacal Nitrogen present in the given sample is----- mg/L

Ex No: 16

DETERMINATION OF COD

Date:

Aim:

To determine the amount of Chemical Oxygen Demand present in the given sample.

Principle:

COD test is widely used for measuring the pollution strength of waste water. All organic compounds with a few exceptions can be oxidized to CO₂ and water by the action of strong oxidizing agents regardless of biological assimilability of the substances.

Apparatus Required:

1. COD Reactor
2. Burette with stand
3. Pipette
4. Measuring jar
5. Tiles
6. Beakers
7. Conical flask

Chemicals Required:

1. Std. Potassium dichromate
2. Conc. Sulphuric acid
3. Ferroin indicator solution
4. Std. Ferrous ammonium sulphate solution
5. Mercuric Sulphate

Reagents Preparation:

Std. Potassium dichromate (0.25 N) solution:

Dissolve 12.26 gm of Potassium dichromate previously dried at 180°C for 2 hr in distilled water and diluted to 1 litre.

Ferroin Indicator Solution:

Dissolve 1.485 gm of 1, 10 Phenolphtholine sulphate monohydrate with 0.695gm of ferrous Sulphate (FeSO₄.7H₂O) in water and dilute to 100ml.

Std. Ferrous Ammonium Sulphate 0.25N:

Dissolve 98gm FAS in distilled water and add 20ml of Conc. H₂SO₄. Cool and dilute to 1 litre. This solution must be standardized against the K₂Cr₂O₇ every day of its use.

Procedure:

1. Take 50ml of sample in a flask and add boiling chips and 1gm of HgSO_4 and 5 ml of H_2SO_4 add slowly to dissolve HgSO_4 and cool the mixture.
2. Add 25 ml of 0.25N $\text{K}_2\text{Cr}_2\text{O}_7$ solution and gain mix. Attach the condense and start the cooling water. The remaining acid agent is added thoroughly through the open end of condenser and the efflux mixture was mixed. Apply the heat and reflux for 2hrs.
3. Dilute the mixture to about 300ml and titrate excess dichromate with std.FAS using Ferro in indicator.
4. The colour will change from yellow to green to blue and finally red and the ml of titrate was deduced.
5. Reflux on the same manner to a flask consisting of distilled water, equal to the volume of the sample and the reagents titrate as he sample and ml of titrant was deduced.

Environmental Significance:

1. BOD cannot be determined accurately when toxins are present and conditions are un favour for the growth of microbes.
2. BOD test consumes more time i.e a minimum of 5 days where COD test is relatively faster than BOD taking only 3hr for completion.

Application of COD:

1. COD test used extensively in the analysis of industrial wastes.
2. It is particularly valuable in survey system to determine and control losses in sewer system.
3. This test is widely used I BOD in the operation of treatment facilities because of the speed with which the result can be obtained.
4. It is useful to access the strength of waste which contains toxins and biological resultant and organic substance.
5. The ratio of BOD to COD is useful to Access the amenability of waste for biological treatment.
6. The ratio of BOD to COD is greater than or equal to 0.8 indicates that the waste water are highly amenable to biological treatment.