Name of the Student : M. Ravi Kiran reddy

Class/Year/Semester/Section : III - II

Registration Number : 19095A0150

INDEX

Sr. No	DATE	NAME OF THE EXPERIMENT	PAGE NO	MARKS
1	17/8/21	Determination of pH value of the water sample	1	10
2		Determination of Turbidity of the water sample		
3		Preliminary Examination of water (a) Taste (b) Odour		
4	17/8/21	a) Determination of the Carbonate, Bicarbonate and Hydroxide Alkalinity b) Determination of the type and extent of Acidity	15	10
5	17/8/21	Determination of Electrical Conductivity of the water sample	23	10
6	17/8/21	Determination of Total Dissolved Solids of the water sample	25	10
7		Estimation of the Chloride Concentration		
8	17/8/21	Estimation of Dissolved Oxygen	31	10
9		Estimation of Hardness of water by EDTA Titration Method		
10		Estimation of the optimum alum dose for coagulation(jar test)		
11		Determination of Nitrogen (Nitrate)		
12		Estimate of percentage of Biochemical Oxygen Demand (BOD)		
13		Estimate the percentage of Chemical Oxygen Demand (COD)		
14		Determination of Total solids, Suspended solids and dissolved solids		
15		Determination of Available chlorine in (a)Residual Chlorine..... (b) Bleaching powder.....		

(ESTD-1995)

SCHOOL OF CIVIL ENGINEERING

RAJEEV GANDHI MEMORIAL COLLEGE OF ENGINEERING & TECHNOLOGY [Autonomous]

NANDYAL – 518501 (AP)

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Course Outcomes:

At the end of the course student is able to

- Determine Physical, Chemical and Biological characteristics of water and wastewater.
- Determine Optimum dosage of Coagulants.
- Asses the Quality of water and wastewater.
- Capable to operate Environmental testing Equipments

1. List of Equipment in Environmental Engineering Laboratory

S.NO	LIST OF EQUIPMENT
1	Electronic Weight Balance
2	BOD Incubator
3	Oven
4	Digital pH meter
5	Digital Conductivity meter
6	UV Visible Spectrophotometer
7	Digital TDS meter
8	Magnetic Stirrer
9	COD Digester
10	Digital DO Meter
11	Jar Test Apparatus
12	Muffle Furnace
13	Colorimeter
14	Nephelometric Turbidity Meter
15	Distillation Apparatus
16	Simple Balance
17	Digital Sound Level Meter

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

2. Drinking water standards of BIS (IS: 10500:2012)

Sr. No.	Parameters	Desirable limit (mg/l)	Permissible limit (mg/l)
Organoleptic and Physical Parameters			
01	Colour Hazen unit, Max	5	15
02	Odour	Agreeable	
03	Taste	Agreeable	
04	Turbidity (NTU*), Max	1	5
05	pH	6.5 – 8	No relaxation
06	Total Dissolved Solids, Max	500	2000
General Parameters Concerning Substances Undesirable in Excessive Amounts			
01	Aluminium (as Al), Max	0.03	0.2
02	Ammonia (as total ammonia-N), Max	0.5	No relaxation
03	Anionic detergents (as MBAS), Max	0.2	1.0
04	Barium (as Ba), Max	0.7	No relaxation
05	Boron (as B), Max	0.5	1.0
06	Calcium (as Ca), Max	75	200
07	Chloramines (as Cl2), Max	4.0	No relaxation
08	Chloride (as Cl), Max	250	1000
09	Copper (as Cu), Max	0.05	1.5
10	Fluoride (as F), Max	1.0	1.5
11	Free residual chlorine, Min	0.2	1
12	Iron (as Fe), Max	0.3	No relaxation
13	Magnesium (as Mg), Max	30	100
14	Manganese (as Mn), Max	0.1	0.3
15	Mineral oil, Max	0.5	No relaxation
16	Nitrate (as NO3), Max	45	No relaxation
17	Phenolic compounds (as C6H5OH), Max	0.001	0.002
18	Selenium (as Se), Max	0.01	No relaxation

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Date
17/08/2021

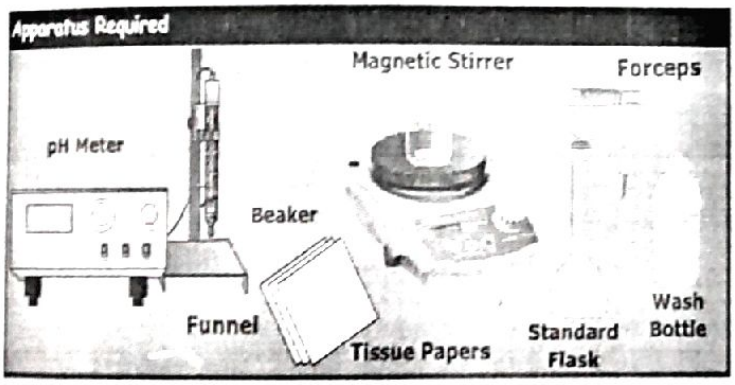
EXPERIMENT No: 1
DETERMINATION OF PH

AIM :-

To determine the PH of the given water sample

APPARATUS REQUIRED

1. pH meter
2. Standard flasks
3. Magnetic Stirrer
4. Funnel
5. Beaker
6. Wash Bottle
7. Tissue Paper
8. Forceps



CHEMICALS REQUIRED

1. Buffers Solutions of pH 4.0, 7.0 and 9.2
2. Potassium Chloride
3. Distilled Water

INTRODUCTION

The term pH refers to the measure of hydrogen ion concentration in a solution and defined as the negative log of H+ ions concentration in water and wastewater. The values of pH 0 to a little less than 7 are termed as acidic and the values of pH a little above 7 to 14 are termed as basic. When the concentration of H+ and OH– ions are equal then it is termed as neutral pH.

PRINCIPLE

The pH electrode used in the pH measurement is a combined glass electrode. It consists of sensing half cell and reference half cell, together form an electrode system.

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

The sensing half cell is a thin pH sensitive semi permeable membrane, separating two solutions, viz., the outer solution, the sample to be analyzed and the internal solution enclosed inside the glass membrane and has a known pH value. An electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the pH of the sample.

ENVIRONMENTAL SIGNIFICANCE:

Determination of pH is one of the important objectives in biological treatment of the wastewater. In anaerobic 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 wastewater. In these circumstances, the pH is generally adjusted by addition of suitable acid or alkali to optimize the treatment of the wastewater. pH value or range is of immense importance for any chemical reaction. A chemical shall be highly effective at a particular pH. Chemical coagulation, disinfection, water softening and corrosion control are governed by pH adjustment.

Dewatering of sludges, oxidation of cyanides and reduction of hexavalent chromium into trivalent chromium also need a favorable pH range. It is used in the calculation of carbonate, bicarbonate, CO2 corrosion, stability index and acid base equilibrium.

Lower value of pH below 4 will produce sour taste and higher value above 8.5 a bitter taste. Higher values of pH hasten the scale formation in water heating apparatus and also reduce the germicidal potential of chlorine. High pH induces the formation of trihalomethanes, which are causing cancer in human beings.

SAMPLE HANDLING AND PRESERVATION:

- ✓ Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.
- ✓ The characteristics of the water sample may change.
- ✓ To reduce the change in samples taken for the determination of pH, keep samples at 40 C. Do not allow the samples to freeze.
- ✓ Analysis should begin as soon as possible.

PRECAUTIONS:

The following precautions should be observed while performing the experiment:

i) Temperature affects the measurement of pH at two points. The first is caused by th change in electrode output at different temperatures. This interference can be controlle by the instruments having temperature compensation or by calibrating the electrod instrument system at the temperature of the samples. The second is the change of p inherent in the sample at different temperatures. This type of error is sample depende and cannot be controlled; hence both the pH and temperature at the time of analysis shou be noted.

ii) In general, the glass electrode is not subject to solution interference like color, hig salinity, colloidal matter, oxidants, turbidity or reductants.

iii) Oil and grease, if present in the electrode

detergent washing, followed by rinsing with distilled water, because it could impair the electrode response.

iv) Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.

v) Electrodes used in the pH meter are highly fragile, hence handle it carefully.

PROCEDURE

Three major steps are involved in the experiment. They are:

1. Preparation of Reagents
2. Calibrating the Instrument
3. Testing of Sample

PREPARATION OF REAGENTS

1. Buffer Solution of pH 4.0

- Take 100 ml standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one buffer tablet of pH 4.0 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 ml using distilled water.

2. Buffer Solution of pH 7.0

- Take 100 ml standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one buffer tablet of pH 7.0 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 ml using distilled water.

3. Buffer Solution of pH 9.2

- Take 100 ml standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one Buffer tablet of pH 9.2 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 ml using distilled water.

CALIBRATING THE INSTRUMENT

Using the buffer solutions calibrate the instrument.

Step 1

In a 100 ml beaker take pH 7.0 buffer solution and place it in a magnetic stirrer, insert the Teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 7.0, using the calibration knob adjust the reading to 7.0.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Step 2

In a 100 ml beaker take pH 4.0 buffer solution and place it in a magnetic stirrer, insert the Teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 4.0, using the slope knob adjust the reading to 4.0.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Step 3

In a 100 ml beaker take pH 9.2 buffer solution and place it in a magnetic stirrer, insert the Teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 9.2, using the slope knob adjust the reading to 9.2.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Now the instrument is calibrated.

TESTING OF SAMPLE

- In a clean dry 100 mL beaker take the water sample and place it on a magnetic stirrer, insert the teflon coated stirring bar and stir well.

- Now place the electrode in the beaker containing the water sample and check for the reading in the pH meter. Wait until you get a stable reading.

- The pH of the given water sample is given

- Take the electrode from the water sample, wash it with distilled water and then wipe gently with soft tissue.

CALCULATION

To determine the value of pH of the given water sample the readings obtained are required to be tabulated

TABLE

Sample No	Sample description	Temperature of Sample (ºC)	PH
1.	River water	27°	8.30
2.	Groundwater	27°	7.45

Drinking water = 6.5 to 8

DATA SHEET

- ✓ Date Tested : 17/8/21
- ✓ Tested By :
- ✓ Sample Number : 1
  Sample Location : SRBC ✓
  Description : Canal / River water

- ✓ Sample Number : 2
  Sample Location :
  Description : Ground water

RESULT:-

The PH of the given sample 1 (Canal / River water) = 8.30

The PH of the given sample 2(Ground water) = 7.45 ✓

CONCLUSION:-

The PH of the most drinking water lies with in the range of 6.5-8.

So, we get pH value of canal and ground water are 8.30 and 7.45. Hence the pH values of water samples are determined.

10

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

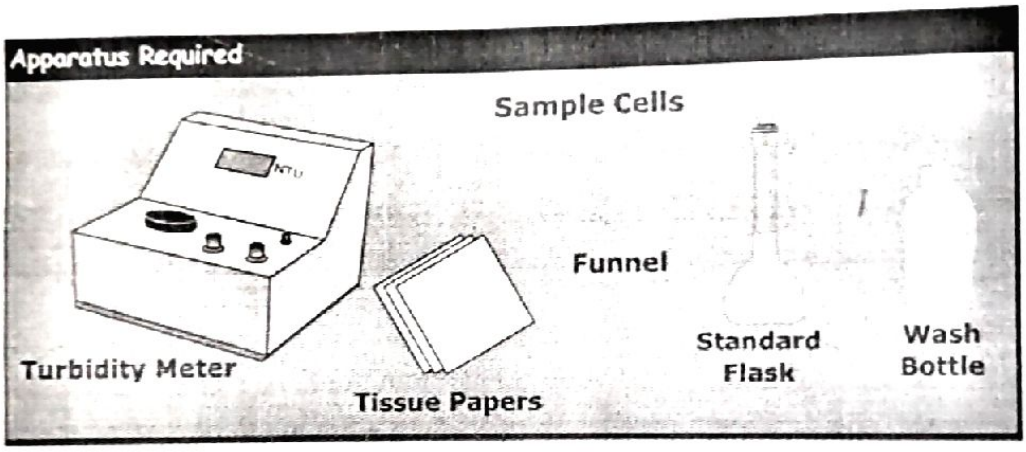
EXPERIMENT No: 2
DETERMINATION OF TURBIDITY

AIM

To determine the Turbidity of the given water sample.

APPARATUS REQUIRED

1. Turbidity Meter
2. Sample Cells
3. Standard flasks
4. Funnel
5. Wash Bottle
6. Tissue Papers



CHEMICALS REQUIRED

1. Hexamethylenetetramine
2. Hydrazine sulphate
3. Distilled water

INTRODUCTION

Turbidity is the technical term referring to the cloudiness of a solution and it is a qualitative characteristic which is imparted by solid particles obstructing the transmittance of light through a water sample. Turbidity often indicates the presence of dispersed and suspended solids like clay, organic matter, silt, algae and other microorganisms.

PRINCIPLE

Turbidity is based on the comparison of the intensity of light scattered by the sample under defined conditions with the intensity of the light scattered by a standard reference suspension under the same conditions. The turbidity of the sample is thus measured from the amount of light scattered by the sample taking a reference with standard turbidity suspension. The higher the intensity of scattered light the higher is the

turbidity. Formazin polymer is used as the primary standard reference suspension.

ENVIRONMENTAL SIGNIFICANCE

When the turbid water in a small, transparent 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 and for biological organism that may not be harmful. They may be harmful or cause undesirable tastes and odours. Disinfection 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 color 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. Knowledge of the turbidity variation in raw water supplies 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. Turbidity measurements are used to determine the effectiveness of treatment produced with different chemicals and the dosages needed. Turbidity measurements help to gauge the amount of chemicals needed from day-to-day operation of water treatment works.

Measurement of turbidity in settled water prior to filtration is useful in controlling chemical dosages so as to prevent excessive loading of rapid sand filters. Turbidity measurements of the filtered water are needed to check on faulty filter operation.

Turbidity measurements are useful to determine the optimum dosage of coagulants to treat domestic and industrial wastewaters. Turbidity determination is used to evaluate the performance of water treatment plants.

SAMPLE HANDLING AND PRESERVATION

Water samples should be collected in plastic cans or glass bottles. All bottles must be cleaned thoroughly and should be rinsed with turbidity free water.

Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

No chemical preservation is required. Keep the samples at 4°C. Do not allow samples to freeze.

Analysis should begin as soon as possible after the collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- The presence of coloured solutes causes measured turbidity values to be low. Precipitation of dissolved constituents (for example, Fe) causes measured turbidity values to be high.
- Light absorbing materials such as activated carbon in significant concentration can

cause low readings.

• The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

PROCEDURE

For testing the given water sample first the reagents are to be prepared. Then the turbidity meter is required to be calibrated.

PREPARATION OF REAGENTS

1. Hydrazine Sulphate $(NH_2)_2H_2SO_4$

• Weigh accurately 1 g of hydrazine sulphate and dissolve it in turbidity free distilled water.

• Take 100 mL standard measuring flask and place a funnel over it.

• Transfer it to a 100 mL standard flask and make up to 100 ml using turbidity free distilled water.

2. Hexamethylene Tetramine $(CH_2)_6N_4$

• Weigh accurately 10 g of Hexamethylene tetramine and dissolve it in turbidity free distilled water.

• Take 100 mL standard measuring flask and place a funnel over it.

• Transfer it to a 100 mL standard flask and make up to 100 ml using turbidity free distilled water.

3. Standard 400 NTU Solution

• Mix 5 mL of hydrazine sulphate solution and 5 mL of Hexamethylenetetramine solution in a 100 mL standard measuring flask.

• Allow the mixture to stand for 24 hours.

• After 24 hours, make up the volume to 100 mL using turbidity free distilled water.

• The standard 400 NTU solution is ready.

solution	suspension	Dist.water
200 JTU	50 ml	50 ml
100 JTU	25 ml	75 ml

CALIBRATION OF TURBIDITY METER

Using the standard solution calibrate the instrument.

1. Switch ON the instrument and keep it ON for some time.
2. Open the lid of the sample compartment. Insert a test tube filled with Distilled water into the sample compartment and close the lid.
3. Rotate button set 0(zero) control to get 0(zero) displayed on the readout.
4. Open the lid and replace the test tube filled with distilled water with test tube filled with (FORMAZINE) standard then close the lid.
5. Rotate button set 100 to get 100 displayed on the readout.
6. Push button set 1000 to get 1000 displayed on the readout.
7. Now the instrument is ready to take measurement of any solution of unknown concentration.

8. Replace the standard test tube with the sample and place the test tube in a sample compartment and close the lid.
9. turbidity value of the sample will be displaced on the readout

TESTING OF WATER SAMPLE

- To the sample cells, add sample water up to the horizontal mark, wipe gently with soft tissue and place it in the turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.
- Check for the reading in the turbidity meter. Wait until you get a stable reading.
- The turbidity of the given water sample is .....NTU.

CALCULATION

For determining the Turbidity of the given water sample the readings are required to be tabulated.

TABLE

Sample No.	Sample Description	Temperature of Sample (ºC)	Turbidity (NTU)
1			
2			
3			

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1
  Sample Location:
  Description: Canal / River water

- ✓ Sample Number :2
  Sample Location:
  Description: Ground water

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

RESULT:-

The Turbidity of the given sample 1 (Canal / River water) =

The Turbidity of the given sample 2 (Ground water) =

CONCLUSION:-

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

EXPERIMENT NO – 03
PRELIMINARY EXAMINATION OF WATER

Experiment 3(A): Taste

AIM: To determine the Flavor Threshold Number of water.

APPARATUS: Flask, Watch glass.

INTRODUCTION:

The "Flavor Threshold Number" (FTN) is calculated corresponding to the greatest dilution of the sample with taste-free water yielding a definitely perceptible change in taste. FTN is defined as:

$$FTN = \frac{A + B}{A}$$

Where: A = ml of sample
B = ml of taste free water

PROCEDURE:

- A sample of water was diluted as shown in the table below and eight aliquots of different dilutions were prepared.
- Taste the individual samples in ascending order starting from sample no 8.
- Determine the sample for which a perceptible taste is first noticed.
- Report the corresponding FTN.

OBSERVATIONS AND CALCULATIONS:

$$FTN = \frac{A + B}{A}$$

Where: A = ml of sample
B = ml of taste free water

Sample No	Sample Volume ml	Sample Volume ml	Diluents Volume ml	Diluents Volume ml	FTN	FTN
1	200		0		1	
2	100		100		2	
3	50		150		4	
4	25		175		8	
5	12		188		17	
6	6		194		33	
7	3		197		67	
8	2		198		100	

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

1.7.2 DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number :
- ✓ Sample Location :
- ✓ Description : Canal /River Water

- ✓ Sample Number : 2
- ✓ Sample Location :
- ✓ Description : Ground Water

RESULTS:

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Experiment 3(B): Odour

AIM: To determine the Threshold Odour Number of water.

APPARATUS: Flask, Watch glass

INTRODUCTION:

The "Threshold Odour Number" (TON) is calculated corresponding to the greatest dilution of the sample with odor-free water yielding a definitely perceptible odor.

TON is defined as:

$$TON = \frac{A + B}{A}$$

Where: A = ml of sample
B = ml of odour free water

PROCEDURE:

- A sample of water was diluted as shown in the table below. The eight aliquots of different dilutions were prepared.
- Taste the individual samples in ascending order starting from sample No. 8.
- Determine the sample for which a perceptible odor is first noticed.
- Report the corresponding TON.

OBSERVATIONS AND CALCULATIONS:

$$TON = \frac{A + B}{A}$$

Where: A = ml of sample
B = ml of odour free water

Sample No	Sample Volume ml	Sample Volume ml	Diluents Volume ml	Diluents Volume ml	TON	TON
1	200		0		1	
2	100		100		2	
3	50		150		4	
4	25		175		8	
5	12		188		17	
6	6		194		33	
7	3		197		67	
8	2		198		100	

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location :
- ✓ Description : Canal /River Water

- ➢ Sample Number : 2
- ➢ Sample Location :
- ➢ Description : Ground Water

RESULTS:

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

17/08/2021

EXPERIMENT NO – 04

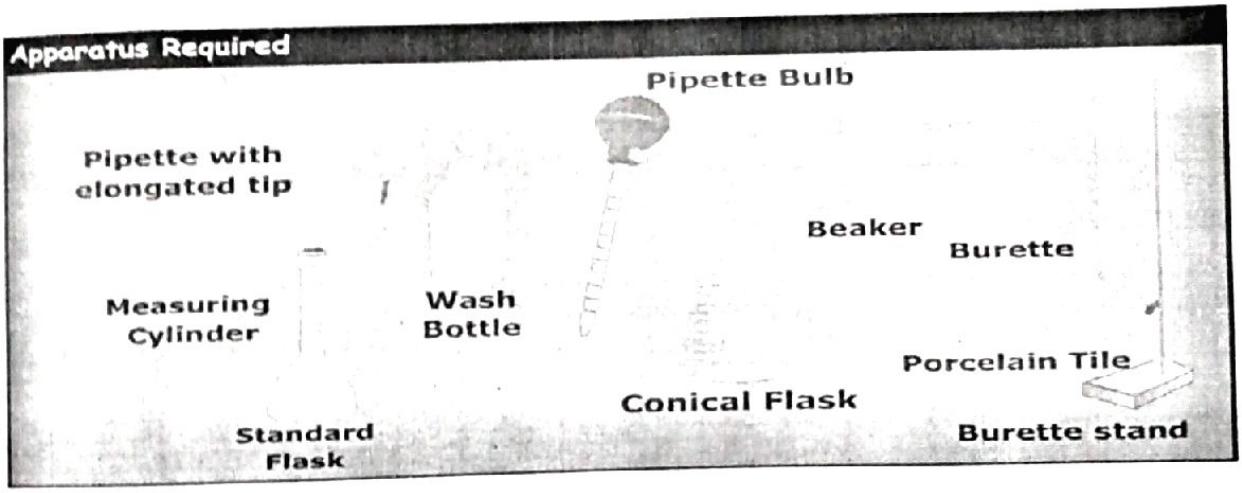
A) Determination of the Carbonate, Bicarbonate and Hydroxide Alkalinity

AIM: To determine various types of alkalinity present in waste water sample

MATERIALS REQUIRED

APPARATUS REQUIRED

1. Burette with Burette stand and porcelain title
2. Pipettes with elongated tips
3. Pipette bulb
4. Conical flask (Erlenmeyer Flask)
5. 250 mL Measuring cylinders
6. Standard flask
7. Wash Bottle
8. Beakers



CHEMICALS REQUIRED

1. Standard sulphuric acid
2. Phenolphthalein
3. Mixed Indicator
4. Bromocresol Green
5. Methyl Red
6. Ethyl alcohol
7. Distilled Water

THEORY:

Alkalinity of waters and wastewaters is the capacity to neutralize acids. In natural water the alkalinity is due to the presence of hydroxide (OH-), carbonate (CO3 2-), and bicarbonates (HCO3 -). Bicarbonates present the major form since they are form inconsiderable amounts from the action of CO2 upon basic materials in the soil.

Head 15

School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

$$CO_2 + CaCO_3 + H_2O \_ Ca(HCO_3)_2$$

Neutral waters may also contain appreciable amounts of carbonates and hydroxide alkalinities, particularly surface waters blooming with algae. The algae take up CO2 for its photosynthetic activities and raise the pH. The carbonate alkalinity may be present with either hydroxide or bicarbonate alkalinity, but hydroxide and bicarbonate alkalinity cannot be present in the sample. The total alkalinity of water is determined by titration with a strong acid to Methyl Orange and end point (pH 4.5) and expressed in $CaCO_3$ scale. The relative (HCO_3^-), (CO_3^{2-}), (OH^-) alkalinities are fixed by the pH of water.

ENVIRONMENTAL SIGNIFICANCE

Alkalinity is important for fish and aquatic life because it protects or buffers against rapid pH changes. Higher alkalinity levels in surface waters will buffer acid rain and other acid wastes and prevent pH changes that are harmful to aquatic life.

Large amount of alkalinity imparts bitter taste in water.

The principal objection of alkaline water is the reactions that can occur between alkalinity and certain cations in waters. The resultant precipitate can corrode pipes and other accessories of water distribution systems.

Wastewaters containing excess caustic (hydroxide) alkalinity are not to be discharged into natural water bodies or sewers.

Alkalinity as carbonate and bicarbonate of saline water is very important in tertiary recovery processes for recovering petroleum. Alkaline water offers better wetting to the formation rock and improve oil release. As an additional benefit, ions that provide alkalinity absorb on rock surfaces occupying adsorption sites and decrease the loss of recovery chemical by adsorption.

The alkalinity value is necessary in the calculation of carbonate scaling tendencies of saline waters.

The alkalinity acts as a pH buffer in coagulation and lime-soda softening of water.

In wastewater treatment, alkalinity is an important parameter in determining the amenability of wastes to the treatment process and control of processes such as anaerobic digestion, where bicarbonate alkalinity, total alkalinity, and any fraction contributed by volatile acid salts become considerations.

SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

To reduce the change in samples, keep all samples at 4°C. Do not allow samples to freeze.

Analysis should begin as soon as possible.

Do not open sample bottle before analysis.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

1. Do not keep the indicator solution open since it contains the alcohol which tends to evaporate.
2. The mixed indicator solution is containing dye in it; care should be taken so that it is not spilled to your skin.
3. If it spills on your skin, the scar will remain at least for two to three days.

PREPARATION OF REAGENTS

For testing the given sample, first the reagents are required to be prepared.

Sulphuric Acid Solution (0.02N):

- Take approximately 500 mL of distilled water in a 1000 mL standard flask.
- Pipette 20 mL of concentrated 0.1 Normality Sulphuric acid and add slowly along the sides of the standard flask.
- Then make up the volume up to 1000 mL mark. Now the strength of this solution is 0.02 N.

Phenolphthalein Indicator Preparation:

- Weigh 1g of phenolphthalein and add to 100 mL of 95% ethyl alcohol or to 100 mL of distilled water. Use the readymade Phenolphthalein indicator available in the market.

Methyl orange indicator:

0.5 g of methyl orange powder is dissolved in CO_2 free distilled water (pH 4.3- 4.5) and diluted to 1 liter.

PROCEDURE

Take 100ml of sample in a conical flask. Add 3-4 drops of phenolphthalein indicator. If no colour is produced, the phenolphthalein alkalinity is absent. If the sample turns pink, titrate with N/50 H_2SO_4 till the pink colour disappears. Record the ml of acid used (P). Add 1 drop of methyl orange to same sample to the titrated mixture and retitrate with N/50 H_2SO_4 until first change from yellow to orange colour is noted (T)

Titration using phenolphthalein = P
Titration using methyl orange = T (includes P)
Phenolphthalein alkalinity = (P X 1000)/ml sample
Total alkalinity = (T X 1000)/ml sample mg/l $CaCO_3$

Once, the phenolphthalein and total alkalinities are determined, three types of alkalinities, i.e. hydroxide, carbonate and bicarbonate are easily calculated from the table given as under:

Type of alkalinity

Values of P	Type of Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as	Bicarbonate Alkalinity as
P = 0	0	0	T
P < 1/2T	0	2P	T – 2P
P = 1/2T	0	2P	0
P > 1/2T	2P –T	2(T – P)	0
P = T	T	0	0

Once carbonate and bicarbonate alkalinities are known, then their conversions to milligrams CO_3^{--} or HCO_3^-/L are possible.

mg CO_3^{--}/L = Carbonate alkalinity mg $CaCO_3$/L x 0.6

mg HCO_3 = Bicarbonate alkalinity mg $CaCO_3$/L x 1.22

from above, molar concentration may be obtained as follows: $[CO_3^{--}]$ = mg/L CO_3 / 60000

$[HCO_3^-]$ = mg/L HCO_3 / 61000

Data sheet: determination of alkalinity

Sample detailed source	Volume (ml)	pH	Phenolphthalein (did not change colour)			Methyl Orange		
			Initial	final	Final H_2SO_4 reading	initial	final	Final H_2SO_4 reading
River water	100ml	8.30	—	—	—	0	7.5	7.5ml

Sample	Total alkalinity mg/l $CaCO_3$	OH- alkalinity mg/l	CO_3 2- alkalinity	HCO_3- alkalinity
River water	75 mg/l	0	0	75 mg/l

DATA SHEET

- ✓ Date Tested : 17/08/21
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location : SRBC
- ✓ Description : River water.

$$\frac{0.02 * 7.5 * 1000 * 50}{100} = 75 mg/l$$

Calculations:

Total alkalinity ($CaCO_3$) mg/l = (m/ N/50 H_2SO_4 used X 1000)/ ml of sample

mixed color

RESULTS:

The alkalinity value in river water = 75 mg/l $CaCO_3$.

CONCLUSION: ~~Hence the acidity~~

Alkalinity measures the acid-Neutralization capacity of waste water.

In conclusion we had achieved all the objectives for this experiment. The bore water sample has alkalinity 75 mg/l. Hence the total alkalinity in water sample is determined.

The alkalinity is less than 200 mg/l for drinking purpose

Alkalinity

(0.02N) Sulfuric acid (H_2SO_4) – Burette (Upward)

↓ River water – conical flask (100ml)

① Phenolphthalein – 3 drops

② methyl red – 3 drops

↓

titrate up to pink. [note down readings.

Acidity

Sodium hydroxide.

NaOH – Burette.

↓

River water

↓

methyl → water to organic red-colour

↓

titrate – up to yellow

acidity.

If not

↓ Phenolphthalein

Peal Pink or not

↓

up to titrate → Peal Pink

10

Head

School of Civil Engineering

Rajeev Gandhi Memorial College of Engg. & Tech. (Autonomous)

NANDYAL-518 501.

B) Determination of the type and extent of Acidity

The acidity of natural water is primarily due to dissolved CO_2 and is defined as the capacity to neutralize bases. However in water polluted by trade wastes acidity may be because of because of mineral acids (below pH 4)

PROCEDURE

Place 100 ml water in a conical flask and add to it one drop of Methyl orange indicator. If it gives an orangish red colour, Mineral acidity is present. Titrate it with N/50 NaOH to a yellow end point. Note the ml of N/50 NaOH used.

In another flask place 100 ml water and add 0.5 ml phenolphthalein indicator. If it does not give any color, titrate with N/50 NaOH to light pink (first permanent change) end point. Note the ml of solution used. If Phenolphthalein gives a pink color on addition of the sample, acidity is not available.

CALCULATIONS

Mineral acidity mg/l ($CaCO_3$ scale) = (ml of NaOH used with every Methyl orange) x1000 / ml Sample = 0

CO_2 acidity mg/l ($CaCO_3$) = (ml of NaOH used with phenolphthalein) x1000/ml sample

Sample detailed source	Volume (ml)	Phenolphthalein			Methyl Orange		
		initial	Final	Final NaOH reading	initial	final	Final NaOH reading
River water	100ml	0	1.5	1.50 ml	0	0	0

Note: Acidity due to CO_2 is present within a pH range 4.5-8.3. When the pH of a Sample is more than 8.3, acidity is absent.

DATA SHEET

- ✓ Date Tested: 17/08/21
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location : SRBC
- ✓ Sample Description : River water

$\Rightarrow \frac{1.5 * 1000}{100}$

$\Rightarrow 15 mg/l$

Head

School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

RESULTS: The acidity value in River water = 15 mg/l

The mineral acidity is absent and CO_2 acidity is Present

The mineral acidity is 0

CONCLUSION: Hence the acidity present in the sample of water is determined.

For the construction water sample should be at 50 mg/l for acidity.

9

10

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

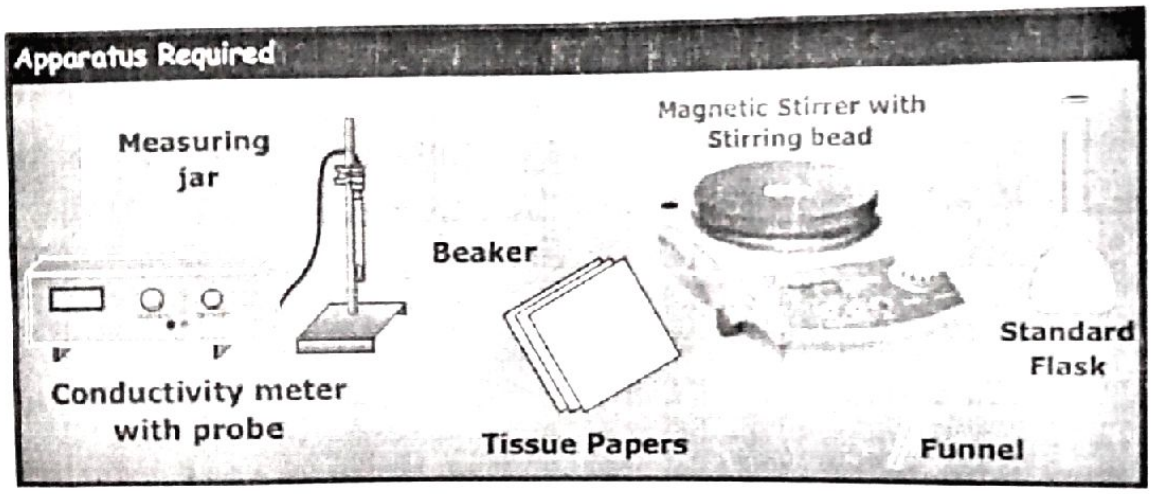
17/08/2021

EXPERIMENT NO – 05
DETERMINATION OF ELETRICAL CONDUCTIVITY

AIM: To determine the Electrical Conductivity of the Water Sample by using Digital Conductivity meter.

APPARATUS REQUIRED:

1. Conductivity Meter with Electrode
2. Magnetic Stirrer with stirring bead
3. Standard flask
4. Measuring jar
5. Beaker 250 mL
6. Funnel
7. Tissue Paper



PRINCIPLE:

This method is used to measure the conductance generated by various ions in the solution/water. Rough estimation of dissolved ionic contents of water sample can be made by multiplying specific conductance (in S/cm) by an empirical factor which may vary from 0.55 to 0.90 depending on the soluble components of water and on the temperature of measurement. Conductivity measurement gives rapid and practical estimate of the variations in the dissolved mineral contents of a water body.

Analytical instrument:

a. Self-contained conductance instruments: (conductivity meter) These are commercially available.

b. Thermometer, capable of being read to the nearest 0.1°c and covering the range 10-50°c.

c. Conductivity Cells: The cell choice will depend on the expected range of conductivity and the resistance range of the instrument. Experimental results with the true conductance of the potassium chloride solution.

ENVIRONMENTAL SIGNIFICANCE

- Electrical conductivity measurements are often employed to monitor desalination plants.

- It is useful to assess the source of pollution.

- In coastal regions, conductivity data can be used to decide the extent of intrusion of sea water into ground water.

- Conductivity data is useful in determining the suitability of water and wastewater for disposal on land. Irrigation waters up to 2 millisiemens / cm conductance have been found to be suitable for irrigation depending on soils and climatic characteristics.

- It is also used indirectly to fine out inorganic dissolved solids.

Reagents and standards:

Conductivity Water: The conductivity of the water should be less than 1mho/cm; standard potassium chloride: 0.01N; dissolve 745.6mg anhydrous KCl in conductivity water and make up to 1,000 ml at 25°c. This is the standard reference solution, which at 25°c has a specific conductance of 1,413mhos/cm. it is satisfactory for most waters when using a cell with a constant between 1 and 2. Store the solutions in glass stoppered Pyrex bottles.

PROCEDURE:

Calibration:

1. Switch ON the instrument half an hour before the conduction of experiment
2. Electrode/probe is to be washed with Distilled water and do not dip in any solution
2. Press ENTER for getting SELECTION MODE. Select mode as (EC) using direction keys (↑ or↓) and presses ENTER.
3. Enter Cell constant value as1.00 (Press ESC key, if cell of different cell constant say 0.5., is being used and enter the cell constant value) and press Enter
4. If automatic temperature meant is desired and thermo probe is not connected in which case connect it and press Enter key to get temperature.
5. If the temperature is desired to be entered manually, press ESC key and enter temperature.
6. If the actual temperature is not what is displayed as default, measure the temperature with a thermometer, press ESC key and enter the temperature.
7. Keep the sample-filled-container near the cell/probe stand and lower the holding clamp to dip the sensor part of the cell in the sample. Insert thermo-probe also into the sample. Press ENTER key and it display cell constant value as 1.00 and then dip the cell in the sample and press Enter. There temperature and EC values are displayed.

DATA SHEET

- ✓ Date Tested: 17/08/21
- ✓ Tested By :
- ✓ Sample Number : 1 Tap water
- ✓ Sample Location :
- ✓ Sample Description : Tapwater
- ✓ Sample Number : 2
- ✓ Sample Location :
- ✓ Sample Description : River water.

RESULT:

- ✓ Electrical conductivity of water sample 1 ..... 0.963 micro siemens/cm [Tap water]
- ✓ Electrical conductivity of water sample 2 ..... 395 µS [River water]

CONCLUSION: Hence the electrical conductivity of water sample is determined by using the digital Conductivity meter. In experiment A the conductivity of the several different Compounds was tested in distilled water. The first few tested were all molecular compounds and exhibited little to no change in Charge at all.

mode
↓
Enter
↓
EC & TDS
↓
enter
↓
Constant [1]
↓
enter
↓
value taken.

10

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

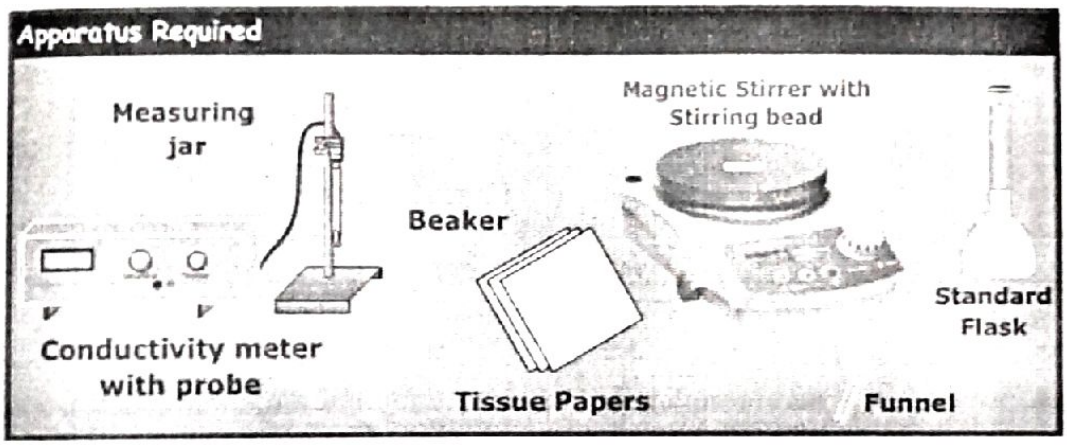
17/08/2021

EXPERIMENT NO – 06
DETERMINATION OF TOTAL DISSOLVED SOLIDS

AIM: To determine the TDS of the Water Sample by using Digital TDS meter

APPARATUS REQUIRED:

1. Conductivity Meter with Electrode
2. Magnetic Stirrer with stirring bead
3. Standard flask
4. Measuring jar
5. Beaker 250 ml
6. Funnel
7. Tissue Paper



INTRODUCTION:

The term 'solid' refers to mater either filterable or non-filterable that residue upon evaporation and subsequent drying at a defined temperature. Further categorization depends upon the temperature employed for drying and ignition. Different forms of solids are defined on the basis of method applied for their determination. Solids may affect water or effluent quality adversely in number of ways. Water with high dissolved solids may induce an unfavorable physiological reaction in the transient consumer and generally are of inferior palatability. Highly mineralized waters are unsuitable for many industrial applications. High suspended solids in waters may be aesthetically unsatisfactory for such purposes as bathing. Analysis of total solids is important to decide upon the various unit operations and processes in physical and biological wastewater treatment and to assess its performance evaluation. For assessing compliance with regulatory agency, wastewater effluent

Limitation for various forms of solid act ac indicating parameters.

PRINCIPLE:

Residue left after the evaporation and subsequent drying in oven at specific temperature 103-105°c of a known volume of sample are total solids. Total solids include "Total suspended solids" (TSS) and "Total dissolved solids" (TDS). Where loss in weight on ignition of the same sample at 500°c ± 50°c, in which organic matter is converted to CO_2

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

and H_2O while at controlled temperature to prevent decomposition and volatilization of inorganic matter as much as consistent with complete oxidation of organic matter, are volatile solids.

PROCEDURE:

Calibration:

1. Switch ON the instrument half an hour before the conduction of experiment
2. Electrode/probe is to be washed with Distilled water and do not dip in any solution
3. Press ENTER for getting SELECTION MODE. Select mode as (TDS) using direction keys (↑ or↓) and presses ENTER.
4. Enter TDS Factor as 0.50. (Press ESC if it is desired to enter a different TDS Factor say 0.56.,) and press Enter
5. Enter Cell constant value as1.00. (Press ESC key, if cell of different cell constant say 0.5., is being used and enter the cell constant value) and press Enter.
6. If automatic temperature meant is desired and thermo probe is not connected in which case connect it and press Enter key to get temperature.
7. If the temperature is desired to be entered manually, press ESC key and enter temperature.
8. If the actual temperature is not what is displayed as default, measure the temperature with a thermometer, press ESC key and enter the temperature.
9. Keep the sample-filled-container near the cell/probe stand and lower the holding clamp to dip the sensor part of the cell in the sample. Insert thermo-probe also into the sample. Press ENTER key and it display cell constant value as 1.00 and then dip the cell in the sample and press Enter. There temperature and TDS values are displayed.

.

DATA SHEET

- ✓ Date Tested:
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location :
- ✓ Sample Description :
- ✓ Sample Number : 2
- ✓ Sample Location :
- ✓ Sample Description :

RESULTS:

PPM (Parts per million)

- ✓ TDS count of the water sample 1 ..... 529 [TAP water]
- ✓ TDS count of the water sample 1 ..... 219 PPM [River water]

CONCLUSION:

Hence the total dissolved solids in water is determined by using the digital TDS meter while titration is an accurate method of Establishing DO, It is really a laboratory tool and will take up valuable time. The TDS is less than 500 PPM for drinking & construction purpose.

9. 10

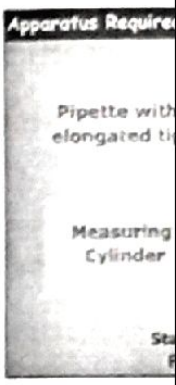
tool

AIM

To det

APPARATU

1. Bur
2. Pip
3. Cor
4. Sta
5. Bea
6. Wa



CHEMICALS

1. Silv
2. Phe
3. Sod
4. Pot

INTRODUCT

Chlor
and wastew
variable and
The r
The salty tas
In sor
salty taste if
taste may b
example 100

This is
calcium or m

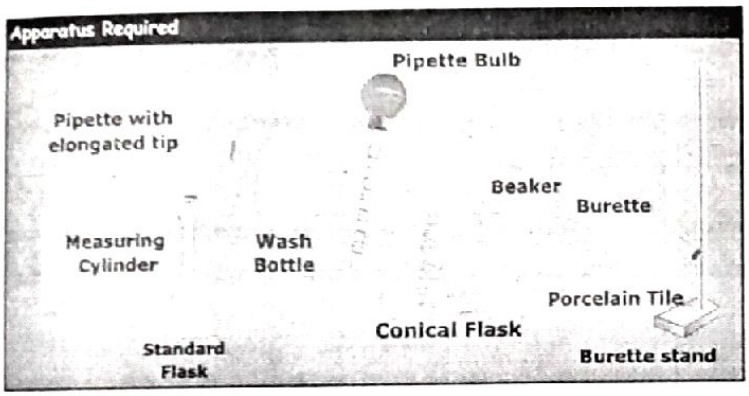
EXPERIMENT NO: 7
ESTIMATION OF CHLORIDE CONCENTRATION

AIM

To determine the chlorides of given water sample

APPARATUS REQUIRED

1. Burette with Burette stand and porcelain tile
2. Pipettes with elongated tips
3. Conical flask (Erlenmeyer Flask)
4. Standard flask
5. Beaker
6. Wash bottle



CHEMICALS REQUIRED

1. Silver nitrate
2. Phenolphthalein Indicator
3. Sodium chloride
4. Potassium chromate

INTRODUCTION

Chlorides are widely distributed as salts of calcium, sodium and potassium in water and wastewater. In potable water, the salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water.

The major taste producing salts in water are sodium chloride and calcium chloride. The salty taste is due to chloride anions and associated cations in water.

In some water which is having only 250 mg /L of chloride may have a detectable salty taste if the cat-ion present in the water is sodium. On the other hand, a typical salty taste may be absent even if the water is having very high chloride concentration for example 1000 mg /L.

This is because the predominant cation present in the water is not sodium but either calcium or magnesium may be present.

PRINCIPLE

The amount of chloride present in water can be easily determined by titrating the given water sample with silver nitrate solution.

The silver nitrate reacts with chloride ion according to1 mole of AgNO3 reacts with 1 mole of chloride. The titrant concentration is generally 0.02 M.

Silver chloride is precipitated quantitatively, before red silver chromate is formed.

The end of titration is indicated by formation of red silver chromate from excess silver nitrate.

The results are expressed in mg/L of chloride (Cl- with a molecular weight of 35.453 g/mol).

ENVIRONMENTAL SIGNIFICANCE

- Chlorides associated with sodium (Sodium Chloride) exert salty taste when its concentration is more than 250 mg/L. These impact a salty taste to water. Chlorides are generally limited to 250 mg/L in water supplies intended for public water supply.

In many areas of the world where water supplies are scarce, sources containing as much as 2000 mg/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. Magnesium chloride in water generates hydrochloric acid after heating which is also highly corrosive and creates problem in boilers.
- Chloride determinations in natural waters are useful in the selection of water supplies for human use.
- Chloride determination is used to determine the type of desalting apparatus to be used.
- Chloride determination is used to control pumping of ground water from locations where intrusion of seawater is a problem.
- Chlorides interfere in the determination of chemical oxygen demand (COD).

SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If Analysis is to be carried out with in two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample 0
collection to reduce the change in sample, keep all samples at 4 C.

Do not allow samples to freeze. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection

PRECAUTIONS

- AgNO3 should be stored in a brown amber bottle and should not be exposed to sunlight.

- While handling $AgNO_3$, care should be taken so that it is not spilled on your skin.
- If it spills on your skin, the scar will remain at least for ten to fifteen days.

PROCEDURE

PREPARATION OF REAGENTS

1. Potassium chromate (K_2CrO_4) indicator
2. N/35.5 Silver nitrate solution

Potassium chromate indicator solution: Dissolve 5 g K_2CrO_4 in about 5 ml distilled water (dH_2O). Add a few drops of silver nitrate until a definite red precipitate forms. Let stand 12 hours, filter and dilute to 100 ml with dH_2O.

Standard sodium chloride (0.0141 N): Dissolve 824.0 mg NaCl (dried at 140°C) in dH_2O and dilute to 500ml. One milliliter of this solution contains how many milligrams of chloride?

Standard silver nitrate titrant (0.0141 N): Dissolve 2.395 g of dry $AgNO_3$ in dH_2O and dilute to 1000 ml. Standardize against 0.0141 N NaCl. One milliliter of this solution will react with how many milligrams of chloride? (1mlof 0.0141N $AgNO_3$ = 0.5 mg Cl)

TESTING OF WATER SAMPLE

- Before starting the titration rinse the burette with silver nitrate solution.

Fill the burette with silver nitrate solution of 0.0141 N. Adjust to zero and fix the burette in stand.

- Take 20 mL of the sample in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of Silver nitrate added **(A).**
- Repeat the procedure for concordant values.

Blank Titration

- Take 20 mL of the distilled water in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of silver nitrate added for distilled water (B).

TABLE

Sample No	Volume of Sample (mL)	Burette Reading (mL) Initial	Final	Volume of $AgNO_3$ (mL)
1				
2				
BLANK				

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Calculation:

Volume of Silver Nitrate for sample (Vs) =
Volume of Silver Nitrate for Blank (VB) =
Normality of Silver Nitrate = N
Volume of Sample =
Equivalent weight of Chlorine = 35.45

Chlorides mg/ L = (Vs – VB) * Normality * 35.45 * 1000 / Volume of sample take
*To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Chlorides mg/ L =

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1
  Sample Location :
  Description : Canal / River water

- ✓ Sample Number :2
  Sample Location :
  Description : Ground water

RESULT:-

Chlorides of the given sample 1 (Canal / River water) =

Chlorides of the given sample 2(Ground water) =

CONCLUSION:-

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

17/08/21

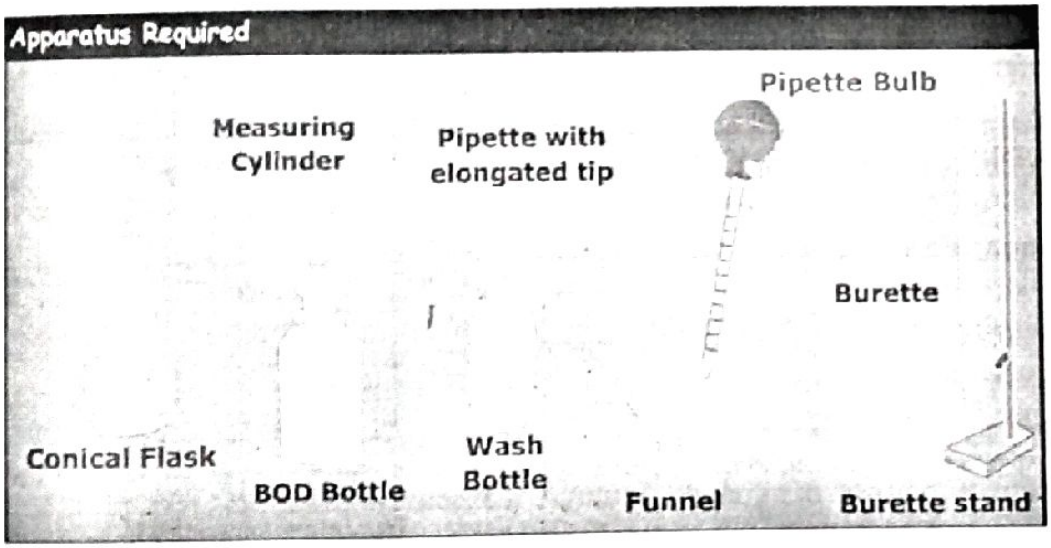
EXPERIMENT No: 8
ESTIMATION OF DISSOLVED OXYGEN

AIM

To determine DISSOLVED OXYGEN (DO) in the given water sample.

APPARATUS REQUIRED

1. Burette
2. Burette stand
3. 300 mL glass stoppered BOD bottles
4. 500 mL conical flask
5. Pipettes with elongated tips
6. Pipette bulb
7. 250 mL graduated cylinders
8. Wash bottle



CHEMICALS REQUIRED

1. Manganous sulphate solution
2. Alkaline iodide-azide solution
3. Sulfuric acid, Concentrated
4. Starch indicator solution
5. Sodium thiosulphate
6. Distilled or deionized water
7. Potassium Hydroxide
8. Potassium Iodide
9. Sodium Azide

INTRODUCTION

Before performing this experiment, few questions may arise to the learners: 1. What

is meant by Dissolved Oxygen (DO)? Is it oxygen in dissolved form?

2. Why we need to determine DO?
3. What are the methods available to determine DO?
4. Is it measured in natural water or wastewater?

5. Whether is it mandatory as per our codal provision to determine DO?

The term Dissolved Oxygen is used to describe the amount of oxygen dissolved in a unit volume of water. Dissolved oxygen (DO) is essential for the maintenance of healthy lakes and rivers. It is a measure of the ability of water to sustain aquatic life.

The dissolved oxygen content of water is influenced by the source, raw water temperature, treatment and chemical or biological processes taking place in the distribution system.

The presence of oxygen in water is a good sign. Depletion of dissolved oxygen in water supplies can encourage the microbial reduction of nitrate to nitrite and sulfate to sulfide. It can also cause an increase in the concentration of ferrous iron in solution, with subsequent discoloration at the tap when the water is aerated.

Hence, analysis of dissolved oxygen is an important step in water pollution control and wastewater treatment process control. There are various methods available to measure Dissolved Oxygen, which we will discuss in detail.

In a healthy body of water such as a lake, river, or stream, the dissolved oxygen is about 8 parts per million. The minimum DO level of 4 to 5 mg/L or ppm is desirable for survival of aquatic life.

Now imagine that a source of oxygen demanding wastes, such as feed lot, a paper mill or a food processing plant, is built besides the river. The facility begins operating and discharging wastes into the river.

This increases the BOD and affects the concentration of DO in the waters downstream.

The wastes serve as the food for certain aerobic bacteria. as it moves downstream, the conc. of bacteria increases. Because these bacteria remove oxygen from water, their population increase causes a decline in the amount of DO.

Beyond certain point, most of the wastes break down. The conc. of DO rises as the river recovers oxygen from the atmosphere and aquatic plants.

Thus DO test is the basis for BOD test which is an important parameter to evaluate organic pollution potential of a waste.

It is necessary for all aerobic biological wastewater treatment processes to control

the rate of aeration.

PRINCIPLE

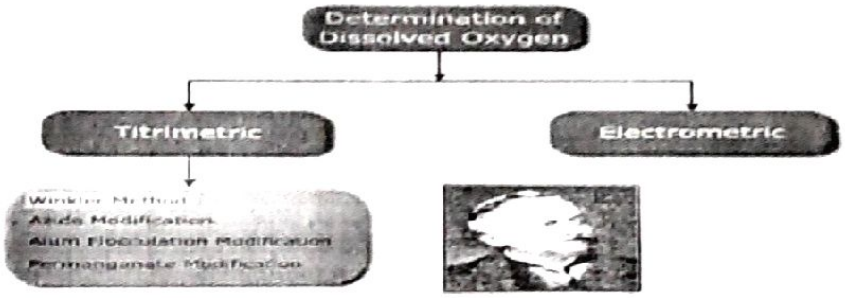
Dissolved Oxygen can be measured either by titrimetric or electrometric method.

(1) Titrimetric Method

Titrimetric method is based on the oxidizing property of DO while the electrometric method (using membrane electrodes) is based on the rate of diffusion of molecular oxygen across a membrane. It is most accurate method to determine DO.

There are different titrimetric methods based on the nature of sample to be tested.

(a) Winkler Method
(b) Azide Modification
(c) Alum Flocculation Modification
(d) Permanganate Modification



However, in all the above the basic principle remains same.

Choice of the method depends upon the type of sample to be tested

Azide Modification:

In this method, interference caused by nitrate is removed effectively. Presence of nitrate is most interference in biologically treated effluent and incubated BOD

Samples.

Alum Flocculation Modification:

If the sample contains suspended solids (especially effluent samples), then this method will be suitable.

Permanganate Modification:

If the sample contains iron (Fe2+) ions. Addition of 1mL of potassium fluoride and azide solution can be adopted to suppress the interference due to (Fe3+

This method is not useful when the sample contains sulphites, thiosulphates and high BOD.

The Titrimetric principle:

Divalent Manganese salt in solution is precipitated by strong alkali to trivalent manganese hydroxide.

Addition of Potassium iodide or Potassium hydroxide is added to create a pinkish brown precipitate.

In the alkaline solution, dissolved oxygen present in the sample rapidly oxidized to form trivalent or higher valency hydroxide.

$MnO(OH)_2$ appears as a brown precipitate. There is some confusion about whether the oxidised manganese is tetravalent or trivalent. Some sources claim that $Mn(OH)_3$ is the brown precipitate, but hydrated MnO_2 may also give the brown colour.

Iodide ions are added and acidified (acid facilitates the conversion by the brown), which reduces tetravalent hydroxides back to their stable divalent state thereby liberating equivalent amount of iodine.

Thiosulphate solution is used, with a starch indicator, to titrate the iodine.

This iodine is equivalent to dissolved oxygen present in the sample.

(2) Electrometric Method

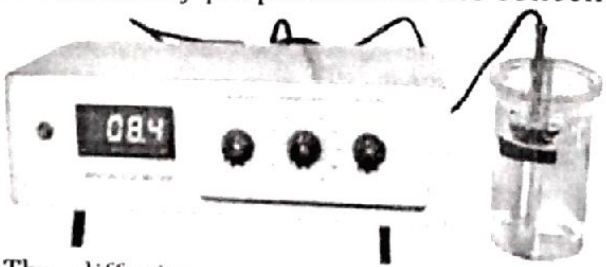
The electrode method offers several advantages over the titrimetric method including speed, elimination or minimization of interferences, field compatibility, continuous monitoring and insitu measurement.

Dissolved oxygen can be measured by a special sensor kept in an electrochemical cell by the amperometric method.

The cell comprises a sensing electrode, a reference electrode and a supporting electrolyte, a semi-permeable membrane, which served dual function.

It separates the water sample from the electrolyte, and at the same time, permits only the dissolved oxygen to diffuse from the water sample through the membrane into the supporting electrolyte.

The diffusion current created by migration of oxygen through a permeable membrane is linearly proportional to the concentration of molecular oxygen in the sample.



The diffusion current created by migration of oxygen through a permeable membrane is linearly proportional to the concentration of molecular oxygen in the sample.

The sample is treated with manganous sulphate, alkaline-iodide-azide reagent and finally sulfuric acid. The first two chemicals combine with dissolved oxygen to form a compound which, when acid is added, releases free iodine (from the potassium iodide).

ENVIRONMENTAL SIGNIFICANCE

Drinking water should be rich in dissolved oxygen for good taste. DO test is used to evaluate the pollution strength of domestic and industrial waste. Higher values of DO may cause corrosion of Iron and Steel. Algae growth in water may release oxygen during its photosynthesis and DO may even shoot upto 30 mg/L. Oxygen is poorly soluble in water. Its solubility is about 14.6 for pure water at 0°C under normal atmospheric pressure and it drops to 7 mg/l at 35°C.

Higher temperature, biological impurities, Ammonia, Nitrates, ferrous iron, chemicals such as hydrogen sulphide and organic matter reduce DO values. Aerobic bacteria thrive when oxygen is available in plenty. Aerobic conditions do prevail when sufficient DO is available within water. End products of aerobiosis are stable and are not foul smelling. It is necessary to know DO levels to assess quality of raw water and to keep a check on stream pollution. DO test is the basis for BOD test which is an important parameter to evaluate organic pollution potential of a waste. DO test is necessary for all aerobic biological wastewater treatment processes to control the rate of aeration.

SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If analysis is to be carried out with in two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample collection to reduce the change in sample, keep all sample at 4° C.

Do not allow samples to freeze. Analysis should begin as shown as possible. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection.

PRECAUTIONS

The experiment involves lot of solutions and additions of strong acid and alkali and hence care should be taken.

- Dissolved oxygen concentrations may change drastically depending upon depth, distance, temperature and period of sampling.
- If the sample was obtained by a sampling device of some kind, the water cannot be simply poured into a BOD bottle, since this would cause aeration of the sample. Instead, the sample must be drawn off from a tube located near the bottom of the sampling device. Place the rubber tube into the bottom of the BOD bottle and fill the bottle, again allowing the bottle to overflow.
- For shallow depth use normal water samplers. However for depth greater than 150 cm (5 ft), use Kemmerer Sample Bottles.

In the case of electrode method:

- Membrane-covered electrode systems minimize the interferences often encountered with dropping mercury or rotating platinum electrodes.
- The sensing element is protected by an oxygen permeable membrane, which serves as a diffusion barrier against matrix interference problems.

PROCEDURE:

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENTS

a) Manganous Sulphate Solution

Dissolve Manganese Sulphate

→ 480g of MnSO4.4H2O

(or)

→ 400g of MnSO4.2H2O

(or)

→ 364 g of MnSO4.H2O

In freshly boiled and cooled distilled water, filter the solution and make up to 1000 mL (One litre). In this experiment, we are using Manganese sulphate Mono hydrate,

Take 364 g Manganese sulphate Mono hydrate (MnSO4.H2O) and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer.

The solution should not give blue color by addition of acidified potassium iodide solution and starch.

b) Alkaline Iodide Sodium Azide Solution

To prepare this reagent we are going to mix three different chemicals Dissolve either

→ 500 g of Sodium Hydroxide (or)

→ 700 g of Potassium Hydroxide and

→ 135 g of Sodium Iodide (or)

→ 150 g of Potassium Iodide

To prepare this reagent, take 700 g of Potassium hydroxide and add 150 g of potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000 mL (One litre).

Dissolve 10 g of Sodium Azide (NaN3)in 40 mL of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

c) Sodium Thiosulphate Stock Solution

Weigh approximately 25 g of sodium thiosulphate (Na2S2O35H2O) and dissolve it in boiled distilled water and make up to 1000 mL. Add 1 g of Sodium Hydroxide to preserve it.

d) Starch Indicator

Weigh 2 g of starch and dissolve in 100 mL of hot distilled water. In case if you are going to preserve the starch indicator add 0.2 g of salicylic acid as preservative.

e) sulphuric acid: H2SO4,conc., 1ml is equivalent to about 3ml alkali iodide-azide reagent.

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

f) standard sodium thiosulphate 0.025N: Dilute 250ml stock $Na_2S_2O_3$. Solution to 1000 ml with freshly boiled and cooled distilled water. Add preservative before making up the volume.

TESTING OF SAMPLE

- Take two 300-mL glass stoppered BOD bottle and fill it with sample to be tested. Avoid any kind of bubbling and trapping of air bubbles. Remember – no bubbles!

(Or)

- Take the sample collected from the field. It should be collected in BOD bottle filled upto the rim.

- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.

- Add 2 mL of alkali-iodide-azide reagent in the same manner.

- Squeeze the pipette slowly so no bubbles are introduced via the pipette (The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample).

- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.

- Allow it to settle for sufficient time in order to react completely with oxygen.

- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.
- At this point, the sample is "fixed" and can be stored for up to 8 hours if kept in a cool, dark place.

- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an conical flask.
- Titration needs to be started immediately after the transfer of the contents to conical flask.
- Titrate it against sodium thiosulphate using starch as indicator. (Add 3 - 4 drops of starch indicator solution)

- End point of the titration is first disappearance of the blue color to colorless.

- Note down the volume of sodium thiosulphate solution added which gives the dissolved oxygen in 7.9 mL

- Repeat the titration for concordant values.

CALCULATION

For determining the Dissolved Oxygen (DO) in the given water sample, the readings are required to be tabulated.

TABLE

Trial No.	Temperature (ºC)	Volume of Sample (mL)	Burette Reading (mL) Initial	Final	Volume of Titrant (mL)	Dissolved Oxygen (mg/L)
1	27°	203 ml	0	16.00	16 ml	15.76 mg/l

Dissolved oxygen (mg/l): volume of sodium Thiosulphate*0.2 *1000/volume of sample taken

$$* \frac{16 * 0.2 * 1000}{203} = 15.76 \text{ mg/l}$$

DATA SHEET

- ✓ Date Tested : 17/08/21
- ✓ Tested By :
- ✓ Sample Number : 1
  Sample Location : SRBC
  Description : Canal / River water

- ✓ Sample Number :2
  Sample Location :
  Description : Ground water

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

RESULT:-

The DO of the given sample 1 (Canal / River water) = 15.76 mg/l

The DO of the given sample 2(Ground water) = -

CONCLUSION:-

Hence the dissolved oxygen in the given sample of water is determined.

Healthy water generally have dissolved oxygen concentration above 6.5 - 8 mg/l.

The dissolved oxygen is greater than "4" used for aquatic purpose.

~~* Potassium nitrate (KNO_3)~~

↓

Sodium thiosulphate in burette

↓

BOD bottle [River water] - 300ml.

↓ manganous sulphate $MnSO_4$ 2ml and azide solution 2ml [for settlement of solids]

↓

H_2SO_4 - 2 ml [for return into its original colour]

↓

203ml taken into Conical flask. by using pipette.

↓

Startch 3 to 4 drops

↓

titrate up to colorless [not dark blue]

(10)

EXPERIMENT NO - 9

Estimation of Hardness of water by EDTA Titration Method

AIM: To determine the total hardness, calcium and magnesium in the given sample.

APPARATUS: 100ml beaker, Burette, 250 ml conical flask, pH meter, 20 ml pipette & simple balance with weights and fractional weights, weighing bottle

REAGENTS: 1. Buffersolution(of ammonia) 2. Eriochrome black 'T' indicator
3. Standard EDTA solution 0.01M 4. Standard Calcium solution
5. Murexide indicator 6.Sodium Hydroxide 2N

THEORY:

- Hardness is caused due the presence of multivalent cations, mainly Ca^{++} and Mg^{++} in water.
- Hard waters have many disadvantages, primarily scale formation (i.e., $CaCO_3$ deposition) and enhanced capacity to precipitate soap. Thus measurement of water hardness is very necessary.
- Total hardness of water is the sum of Ca^{++} and Mg^{++} concentration in water. The results are expressed as calcium carbonate, in mg/L, i.e., "mg/L as $CaCO_3$".
- When total hardness is numerically greater than the sum of carbonate and bicarbonate alkalinity for a water sample, the amount of hardness equivalent to the carbonate plus bicarbonate alkalinity is called "**carbonate hardness**". The amount of hardness in excess of this is called "**noncarbonate hardness**". When hardness numerically is equal to or less than the sum of carbonate and bicarbonate alkalinity, all hardness is carbonate hardness, and noncarbonate hardness is absent.

REAGENT PREPARATION:

- Dissolve 1.179 g of EDTA disodium salt of EDTA dihydrate and 780 mg $MgSO_4.7H_2O$ or 644 mg $MgCl_2.6H_2O$ in 50 mL of distilled water. Add this solution to 16.9 g NH_4Cl and 143 mL conc. NH_4OH with mixing and dilute to 250 mL with distilled water. Store in a plastic bottle. Label, "**Buffer solution for hardness determination**". *(This solution will be available in the laboratory).*
- Dissolve 3.723 g analytical reagent grade EDTA disodium salt dihydrate in distilled water and dilute to 1000 mL. Label, "**EDTA titrant for hardness determination, 0.01 M**". *(This solution will be available in the laboratory).*
- Pour 1.0 g $CaCO_3$ powder in a 500 mL conical flask. Add, a little at a time 1+1 HCl until $CaCO_3$ is dissolved. Add 200 mL of distilled water. Add a few drops of methyl red indicator, and if necessary, adjust the color to orange using acid or base.
- Dilute to 1000mL. Label, "**$CaCO_3$ standard for hardness determination, 1000 mg/L**". *(This solution will be available in the laboratory).*

PROCEDURE:

TOTAL HARDNESS DETERMINATION

- Take 50ml well mixed sample in conical flask
- Add 1-2 ml buffer solution of ammonia
- To each aliquot add a pinch of Eriochrome Black-T powder (indicator) or 2 drops of Eriochrome Black-T. The aliquots are wine-red in color.
- Titrate each aliquot using the standard EDTA (0.01M) solution (in burette).
- At the end point the aliquots change color from wine-red to blue.
- Note down the volume of EDTA required (A)
- Run a reagent blank if buffer is not checked properly. Note the volume of EDTA required for blank (B)
- Calculate the volume of EDTA required for sample i.e., (A-B)
- Value B may taken as 0 , if double distilled water and ' A R ' grade chemicals are used.

PROCEDURE FOR CALCIUM HARDNESS DETERMINATION

- Take a 50 mL of sample in conical flask.
- Add 1 mL NaOH solution to raise PH to about 12.0
- Add a pinch of ammonium purpurate (murexide) powder (indicator).
- Titrate using the standard EDTA solution (in burette) until color change occurs from pink to purple.
- Note the volume of EDTA used (C).

CALCULATIONS:

1. Total Hardness (mg/l) as $CaCO_3$ = (A-B) *1000/ ml of sample taken

2. Calcium Hardness (mg/l) as $CaCO_3$ = C *1000/ ml of sample taken

3. Magnesium Hardness (mg/l) as $CaCO_3$ = Total Hardness – Calcium Hardness

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

TABLE:

Identification of sample	volume of sample taken in ml	Burette readings Initial final	EDTA solution used in ml	Hardness (mg/l) as $CaCO_3$
Total hardness				
Calcium harness				
Magnesium Hardness				

DATA SHEET

- ✓ Date Tested:
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location :
- ✓ Sample Description :

- ✓ Sample Number : 2
- ✓ Sample Location :
- ✓ Sample Description :

RESULTS:

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

EXPERIMENT NO - 10
ESTIMATION OF THE OPTIMUM ALUM DOSE FOR COAGULATION

AIM: To estimate the optimum alum dose for coagulation.

APPARATUS: Jar test apparatus, Turbidity meter, pH meter, beakers, pipettes.

THEORY:
As the water varies widely in quality, the optimum dose is determined in practice by trial. Normally a single coagulant is applied, its does being regulated to the minimum amount necessary for rapid and adequate coagulation.

Alum is the commonest coagulant used in water treatment.

The optimum dose is found by the jar test technique.

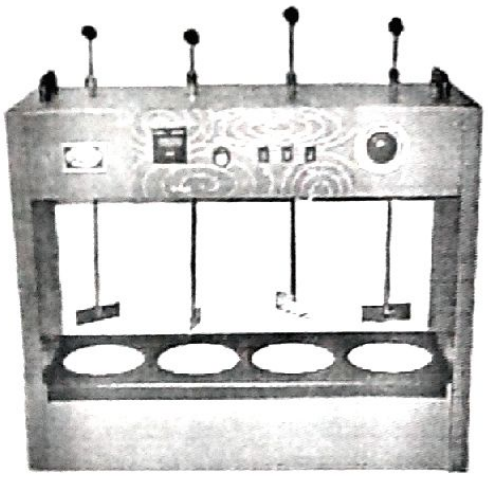
REAGENT
1% alum solution (dissolve 1.0 gram of alum in 100ml distilled water)

ENVIRONMENTAL SIGNIFICANCE:
1. The test is useful for identification of natural coagulants like Nirmali seeds etc
2. it is useful to estimate optimum dosage of coagulant required for raw water and waste waters
3. if excess alum (aluminium sulfate) is used as a coagulat in water treatment plants, aluminium may enter the municipal water and thus the human body. Excess aluminium, if present in drinking water is highly toxic and cause acute abdominal pain and 'dementia '. On the other hand if alum dosage is small, turbidity removal may not be satisfactory.

PROCEDURE
1. Take 1.0 litre of the sample into each of the 6 beakers.
2. Add varying doses of alum solution i.e. 0 ml, 1.0 ml, 2 ml, 3 ml, 4 ml or 6 ml to different beakers simultaneously. One beaker is kept as blank in which no alum is added. These doses correspond to alum doses of 0mg/l, 10 mg/l, 20 mg/l, 30mg/l, 4.mg/l and 60mg/l respectively. The dosage varies with the turbidity of water.
3. Switch on the motor and adjust the speed of paddles to about 100 rpm so that rapid mixing of coagulant is done. Because of flash mixing, the positively charged Aluminium ions will combine with the negatively charged turbidity impurities. Flash mixing (or rapid mixing) is done for 1 to 2 minutes.
4. Reduce the speed of paddles to about 30 to 40 rpm and continue mixing for 10 to 15 minutes. This corresponds to flocculation during which the particles already combined during coagulation combine to form much larger sized agglomerations of floc.
5. Switch off the motor and allow it to settle for 15 to 20 minutes. This corresponds to sediment and find the turbidity of each.

6. Collect the supernatant from each beaker with the help of a pipette without disturbing the sedimentation and find the turbidity of each.

7. Also record pH. Colour, alkalinity and temperature of the supernatant as optimum coagulant dose depend on turbidity of raw water, pH, alkalinity and temperature. Minimum alkalinity required for every 10 mg/l dosage of alum is 4.5 mg/l as $CaCO_3$. Deficiency must be supplemented with lime if the water has alkalinity. Repeat the experiment at different pH values of the samples and at different doses of alum and find the optimum coagulant dose by measuring the turbidity in each case with the help of a nephelo meter or any other turbidity meter. Note the ideal (or optimum) coagulant dose that corresponds to efficient and economical removal turbidity from water.



Data sheet ------ Determination of optimum dose (Alum)

Jar No./ sample detail	ml of 1% alum added	Dosage of alum mg/l	pH	Turbidity NTU

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

DATA SHEET

- ✓ Date Tested:
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location :
- ✓ Sample Description :

RESULTS:

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

EXPERIMENT NO - 11
DETERMINATION OF NITROGEN (NITRATES)

AIM: To find out the amount of Nitrate- nitrogen present in a given sample.

EQUIPMENT AND APPARATUS:

a. Spectrophotometer, for use at 220 nm and 275 nm with matched silica cells of 1 cm or longer light path.
b. Filter: One of the following is required.
   i. Membrane filter: 0.45 µm membrane filter, and appropriate filter assembly
   ii. Paper: Acid-washed, ash less hard-finish filter paper sufficiently retentive for fine precipitates.
c. Nessler tubes, 50 ml, short form.

REAGENTS:

1. Stock Nitrate solution
2. Standard nitrate solution
3. Ammonium hydroxide or KOH

INTRODUCTION:

Determination of nitrate (NO3-) is difficult because of the relatively complex procedure required, the high probability that interfering constituents will be present and the limited concentration ranges of the various techniques. Nitrate is the most highly oxidised form of nitrogen compounds commonly present in natural waters. Significant sources of nitrate are chemical fertilizers, decayed vegetable and animal matter, domestic effluents, sewage sludge disposal to land, industrial discharge, and leachates from refuse dumps and atmospheric washout. Depending on the situation, these sources can contaminate streams, rivers, lakes and ground water. Unpolluted natural water contains minute amounts of nitrate. Excessive concentration in drinking water is considered hazardous for infants because of its reduction to nitrite in intestinal track causing methemoglobinaemia. In surface water, nitrate is a nutrient taken up by plants and converted into cell protein. The growth stimulation of plants, especially of algae may cause objectionable eutrophication.

UV SPECTROPHOTOMETRIC METHOD

The method is useful for the water free from organic contaminants and is most suitable for drinking.

Measurement of the ultraviolet absorption at 220 nm enables rapid determination of nitrate. The nitrate calibration curve follows Beer's law up to 11 mg/L N

Acidification with 1N hydrochloric acid is designed to present interference from hydroxide

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

or carbonate concentrations up to 1,000 mg/L as CaCO3. Chloride has no effect on the determination. Minimum detectable concentration is 40 µg/L NO_3-N.

PRINCIPLE

Nitrate is determined by measuring the absorbance at 220 nm in sample containing 1 ml of hydrochloric acid (1N) in 100 ml sample. The concentration is calculated from graph from standard nitrate solution in range 1-11 mg/L as N.

ENVIRONMENTAL ENGINEERING:

1. Nitrates are converted to nitrites by the nitrate reducing bacteria in the human body. NO2 has greater affinity for oxygen than haemoglobin of blood and hence NO2 gets oxygen from blood to become NO3. Thus, oxygen deficiency occurs and the patient dies of suffocation. As this is common in babies of less than 1 year age and as the death occurs after a bluish discoloration of body this disease is called " BLUE BABY ", or "METHEMOGLOBINEMIA". Blue baby occurs if NO3 is > 45 mg/l. Denitrification process can removes nitrates. It can be found by colorimetric method in the laboratory. Ground water can be contaminated with nitrate by sewage, septic tank effluents and other wastes rich in nitrate.
2. Nitrate determinations are important to know whether the water supplies meet the drinking water standards or not
3. It is used to assess the self purification properties of water bodies and nutrient balance in surface wastes and soil and the state of decomposition of organic matter present in wastewaters.

REAGENTS AND STANDARDS

a. Redistilled water: use redistilled water for the preparation of all solutions and dilutions.
b. Stock nitrate solution: dissolve 721.8 mg anhydrous potassium nitrate and dilute to 1000 ml with distilled water. 1 ml = 100 µg N = 443 µg NO3
c. Standard nitrate solution: dilute 100 ml stock nitrate solution to 1000 ml with distilled water. 1 ml = 10 µg NO3-N = 44.3 µg NO3.
d. Hydrochloric acid solution: HCl, 1N.
e. Aluminum hydroxide suspension: dissolve 125 g potash alum in 1000 ml distilled water. Warm to 60oC, add 55-60 ml NH4OH and allow to stand for 1 h. Decant the supernatant and wash the precipitate a number of times till it is free from Cl, NO2 and NO3. Finally after settling, decant off as much clean liquid as possible, leaving only the concentrated suspension.

CALIBRATION AND STANDARDISATION

Prepare nitrate calibration standards in the range 0 to 350 µg N by diluting 1, 2, 4, 7..... 35 ml of the standard nitrate solution to 50 ml. treat the nitrate standards in the same manner as the samples.

PROCEDURE

Calibration procedure of UV – Spectrophotometer.

1. Switch on the instrument and wait for 10-15 minutes.
2. Select the source as " UV " (or) " VIS " depending on sample
   Clear water (colour less) – UV
   Waste water (colour) - VIS
3. Depending on sample press the source button such that the light makes visible on the source.
4. Select 6th filter " DARK " & then close door
5. Press the " % T " mode
6. Again wait for 10-15 minutes
7. Set Zero (000) using " SETZERO " knob
8. Select wave length by pressing " CLEAR " and then enter wave length of 220(nm) (desirable)
9. Take blank water sample (distilled water) and keep in 1st hole and select 1st filter by taking the filter wheel and close the door.
10. Set the "COARSE "knob to very high.
11. By rotating the "FINE " knob clockwise set to 100
12. Press "ABS" (absorbance) button.
13. Again using the "FINE" knob set to zero (000)
14. Place the sample in the 4th filter hole and note the reading.
15.
16.
17.
18.
19.
20.

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1
  Sample Location :
  Description : Canal / River water/Ground water

RESULT:-

The amount of Nitrate- nitrogen present in the given sample 1 (Canal / River water/Ground water) =

CONCLUSION:-

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

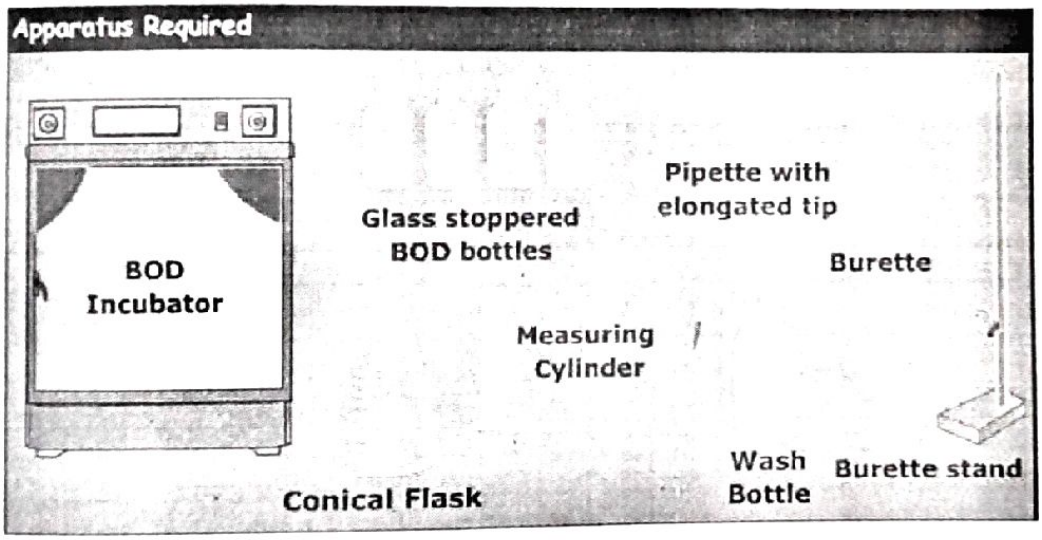
EXPERIMENT No: 12
ESTIMATION OF BIOCHEMICAL OXYGEN DEMAND (BOD)

AIM

To determine biochemical oxygen demand in the given water sample.

APPARATUS REQUIRED

1. BOD Incubator
2. Burette & Burette stand
3. 300 mL glass stopper BOD bottles
4. 500 mL conical flask
5. Pipettes with elongated tips
6. Pipette bulb
7. 250 mL graduated cylinders
8. Wash bottle



CHEMICALS REQUIRED

1. Calcium Chloride
2. Magnesium Sulphate
3. Ferric Chloride
4. Di Potassium Hydrogen Phosphate
5. Potassium Di Hydrogen Phosphate
6. Di sodium hydrogen phosphate
7. Ammonium Chloride
8. Manganous sulphate
9. Potassium hydroxide
10. Potassium iodide
11. Sodium Azide
12. Concentrated sulfuric acid
13. Starch indicator
14. Sodium thiosulphate
15. Distilled or deionized

INTRODUCTION

The biochemical oxygen demand determination is a chemical procedure for determining the amount of dissolved oxygen needed by aerobic organisms in a water body to break the organic materials present in the given water sample at certain temperature over a specific period of time.

BOD of water or polluted water is the amount of oxygen required for the biological decomposition of dissolved organic matter to occur under standard condition at a standardized time and temperature. Usually, the time is taken as 5 days and the temperature is 20°C.

The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulfides and ferrous ion. It also may measure the amount of oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand).

ENVIRONMENTAL SIGNIFICANCE

BOD is the principle test to give an idea of the biodegradability of any sample and strength of the waste. Hence the amount of pollution can be easily measured by it.

Efficiency of any treatment plant can be judged by considering influent BOD and the effluent BOD and so also the organic loading on the unit.

Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants.

Ordinary domestic sewage may have a BOD of 200 mg/L. Any effluent to be discharged into natural bodies of water should have BOD less than 30 mg/L.

This is important parameter to assess the pollution of surface waters and ground waters where contamination occurred due to disposal of domestic and industrial effluents.

Drinking water usually has a BOD of less than 1 mg/L. But, when BOD value reaches 5 mg/L, the water is doubtful in purity.

The determination of BOD is used in studies to measure the self-purification capacity of streams and serves regulatory authorities as a means of checking on the quality of effluents discharged to stream waters.

The determination of the BOD of wastes is useful in the design of treatment facilities.

It is the only parameter, to give an idea of the biodegradability of any sample and self purification capacity of rivers and streams.

The BOD test is among the most important method in sanitary analysis to determine the polluting power, or strength of sewage, industrial wastes or polluted water.

It serves as a measure of the amount of clean diluting water required for the successful disposal of sewage by dilution.

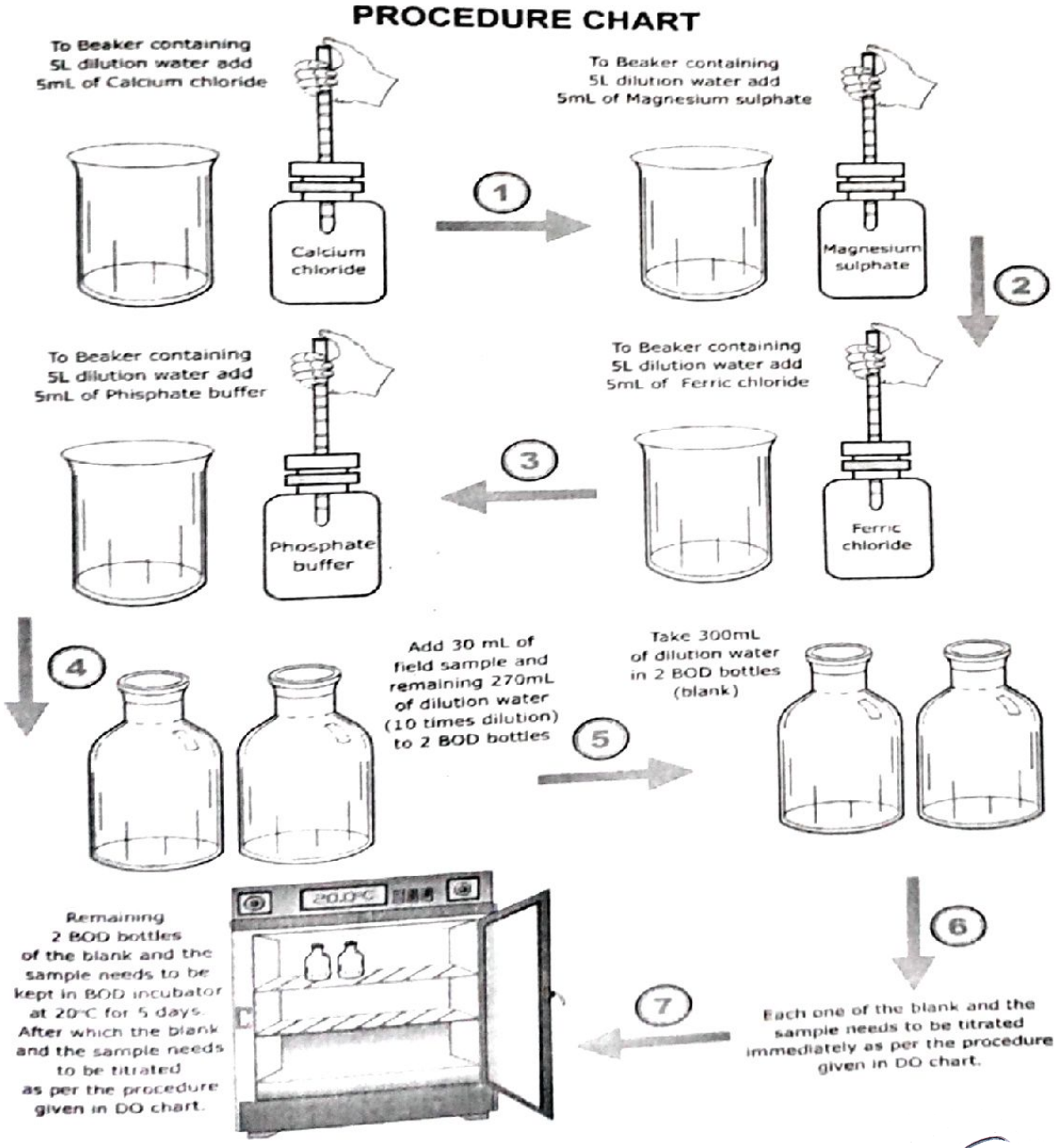
PRINCIPLE

The sample is filled in an airtight bottle and incubated at specific temperature for 5 days.

The dissolved oxygen (DO) content of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between initial and final DO.

The initial DO is determined shortly after the dilution is made; all oxygen uptake occurring after this measurement is included in the BOD measurement.

PROCEDURE CHART



SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If Analysis is to be carried out within two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample collection to reduce the change in sample, keep all samples at 4° C.

Do not allow samples to freeze. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L. **Discard dilution water if there is any sign of biological growth.**
- The sample should be adjusted to a pH between 6.5 and 7.5, using sulfuric acid for samples with pH in the alkaline side i.e., greater than 7.5 or sodium hydroxide for samples with pH in the acidic side i.e., less than 6.5.
- Add sodium sulfite (Na_2SO_3) to remove residual chlorine, if necessary. Samples containing toxic metals, arsenic, or cyanide often require special study and pretreatment.
- While still letting sample water flow down the tube, slowly pull the tube from the bottom of the bottle and fill the bottle to its brim. Check for bubbles. Carefully stopper the BOD bottle as described above.

PROCEDURE

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENT

a) Manganous Sulphate Solution

Dissolve Manganese Sulphate

→ 480g of $MnSO_4.4H_2O$

(or)

→ 400g of $MnSO_4.2H_2O$

(or)

→ 364 g of $MnSO_4.H_2O$

in freshly boiled and cooled distilled water, filter the solution and make up to 1000 mL (One litre). In this experiment, we are using Manganese sulphate Mono hydrate.

Take 364g $MnSO_4.H_2O$ of and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer

Note: The solution should not give blue color by addition of acidified potassium iodide solution and starch.

b) Alkaline Iodide Sodium Azide Solution

To prepare this reagent we are going to mix three different chemicals

Dissolve either

→ 500 g of Sodium Hydroxide (or)
→ 700 g of Potassium Hydroxide
→ 135 g of Sodium Iodide (or)
→ 150 g of Potassium Iodide

To prepare this reagent, take 700 g of potassium hydroxide and add 150 g of potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000 mL (One litre).

Dissolve 10 g of Sodium Azide (NaN_3) in 40 mL of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

c) Sodium Thiosulphate stock solution

Weigh approximately 25 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$)and dissolve it in boiled distilled water and make up to 1000 mL. Add 1 g of sodium hydroxide to preserve it.

d) Starch Indicator

Weigh approximately 2 g of starch and dissolve in 100 mL of hot distilled water.

In case if you are going to preserve the starch indicator add 0.2 g of salicyclic acid as preservative.

e) Sulphuric Acid

add 10 g of Ag_2SO_4 to 1000 ml concentrated H_2SO_4 and let stand for one to two days for complete dissolution.

f) Calcium Chloride solution

Weigh accurately 27.5 g of anhydrous calcium chloride and dissolve it in distilled water.

Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

g) Magnesium Sulphate solution

Weigh accurately 22.5 g of magnesium sulphate and dissolve it in distilled water.

Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

h) Ferric Chloride solution

Weigh accurately 0.15 g ferric chloride and dissolve it in distilled water.

Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

i) Phosphate buffer solution

Weigh accurately 8.5g of Potassium Di Hydrogen Phosphate (KH_2PO_4) and dissolve it in distilled water.

Then add exactly 21.75 g of Di Potassium Hydrogen Phosphate (K_2HPO_4) and dissolve it.

To the same beaker 33.4 g of Di sodium hydrogen phosphate (Na_2HPO_4 $7H_2O$), is weighed and added.

Finally to the beaker containing all the salts, add accurately 1.7 g of Ammonium Chloride (NH_4Cl) and dissolve it.

Take 1000 mL standard measuring flask and place a funnel over it.

Transfer it to the 1000 mL standard flask and make up to 1000 mL using distilled water.

The pH should be 7.2 without further adjustment.

j) Dilution Water

High quality organic free water must be used for dilution purposes.

The required volume of water (five litres of organic free distilled water) is aerated with a supply of clean compressed air for at least 12 hours. Allow it to stabilize by incubating it at 20ºC for at least 4 hours.

For the test we have taken five litres of organic free aerated distilled water, hence add 5mL each of the nutrients.

- Add 5mL calcium chloride solution
- Add 5mL magnesium sulphate solution
- Add 5mL ferric chloride solution and
- Add 5mL phosphate buffer solution

This is the standard dilution water. Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L.

TESTING OF SAMPLE

- Take four 300 mL glass stoppered BOD bottles (two for the sample and two for the blank).
- Add 10 mL of the sample to each of the two BOD bottles and the fill the remaining quantity with the dilution water. i.e., we have diluted the sample 30 times.
- The remaining two BOD bottles are for blank, to these bottles add dilution water alone.
- After the addition immediately place the glass stopper over the BOD bottles and note down the numbers of the bottle for identification.
- Now preserve one blank solution bottle and one sample solution bottle in a BOD incubator at 20ºC for five days.
- The other two bottles (one blank and one sample) needs to be analysed immediately.

Avoid any kind of bubbling and trapping of air bubbles. Remember – no bubbles!

- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- (The pipette should be dipped inside the sample while adding the above two

reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample.)

- Allow it to settle for sufficient time in order to react completely with oxygen.
- When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.
- Titration needs to be started immediately after the transfer of the contents to Erlenmeyer flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an Erlenmeyer flask.
- Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)

Add 1 mL of starch solution.

- and continue the titration until the blue color disappears to colourless.
- Note down the volume of sodium thiosulphate solution added, which gives the D.O. in mg/L. Repeat the titration for concordant values.
- After five days, take out the bottles from the BOD incubator and analyse the sample and the blank for DO.
- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
- Allow it to settle for sufficient time in order to react completely with oxygen.
- When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.
- Titration needs to be started immediately after the transfer of the contents to Erlenmeyer flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an Erlenmeyer flask.
- Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 mL of starch solution and continue the titration until the blue color disappears to colourless.
- Note down the volume of sodium thiosulphate solution added, which gives the D.O. in mg/L. Repeat the titration for concordant values.

CALCULATION

For determining the Biochemical Oxygen Demand in the given water sample, the readings should be tabulated.

TABLE

Trial No.	Day	Volume of Sample (mL)	Burette Reading(mL) Initial Final	Volume of Titrant (mL) ($Na_2S_2O_3$ solution used)	Dissolved Oxygen (mg/L)
Blank					
Blank					

Calculation:

Initial DO of the diluted sample, D0 =

DO at the end of 5 days for the diluted sample, D5 =

Initial DO of the blank, C0 =

DO at the end of 5 days for the blank, C5 =

Blank correction = C0 - C5, BC =

Biochemical Oxygen Demand = {D0– D5 – BC} x Volume of the diluted sample / Volume of sample taken

Biochemical Oxygen Demand (mg/L) =

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1

  Sample Location :

  Description :

RESULT:-

Biochemical Oxygen Demand for the given sample( )=

CONCLUSION:-

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

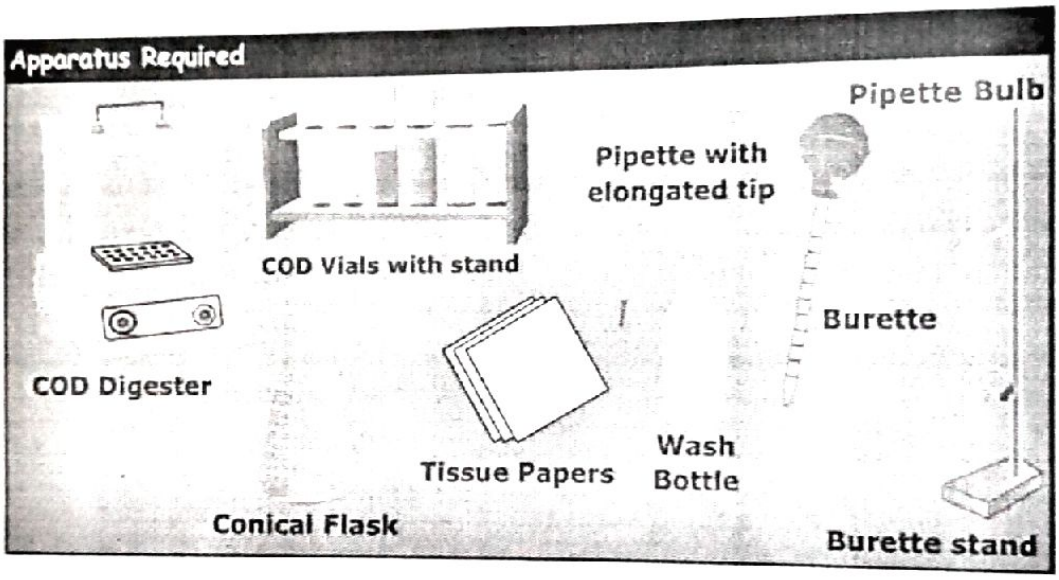
EXPERIMENT NO-13
DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD)

AIM

To determine chemical oxygen demand in the given water sample.

APPARATUS REQUIRED

1. COD Digester
2. Burette & Burette stand
3. COD Vials with stand
4. 250 mL conical flask (Erlenmeyer Flask)
5. Pipettes
6. Pipette bulb
7. Tissue papers
8. Wash Bottle



CHEMICALS REQUIRED

1. Potassium dichromate
2. Sulfuric acid
3. Ferrous ammonium sulphate
4. Silver sulphate
5. Mercury sulphate
6. Ferroin indicator
7. Organic free distilled water

ENVIRONMENTAL SIGNIFICANCE

COD values are particularly important in the surveys designed to determine and control the losses to sewer systems.

The ratio of BOD to COD is useful to assess the amenability of waste for biological

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

treatment. Ratio of BOD to COD greater than or equal to 0.8 indicates that wastewater highly polluted and amenable to the biological treatment.

It is useful to assess strength of wastes, which contain toxins and biologically resistant organic substances.

COD can be related to TOC, however, does not account for oxidation state of the organic matter.

BOD value is always lower than COD value. For domestic and some industrial wastewater, COD value is about 2.5 times BOD value.

PRINCIPLE

The organic matter present in sample gets oxidized completely by potassium dichromate ($K_2Cr_2O_7$) in the presence of sulphuric acid (H_2SO_4), silver sulphate ($AgSO_4$) and mercury sulphate ($HgSO_4$) to produce CO_2 and H2O. The sample is refluxed with a known amount of potassium dichromate ($K_2Cr_2O_7$) in the sulphuric acid medium and the excess potassium dichromate ($K_2Cr_2O_7$) is determined by titration against ferrous ammonium sulphate, using ferroin as an indicator. The dichromate consumed by the sample is equivalent to the amount of O_2 required to oxidize the organic matter.

SAMPLE HANDLING AND PRESERVATION

Samples are collected in glass bottles. Use of plastic containers is permitted if it is known that there is no organic contaminants present in it.

Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.

Samples should be preserved with sulphuric acid to a pH < 2 and maintained at 0

4 C until analysis. Do not allow the samples to freeze.

PRECAUTIONS

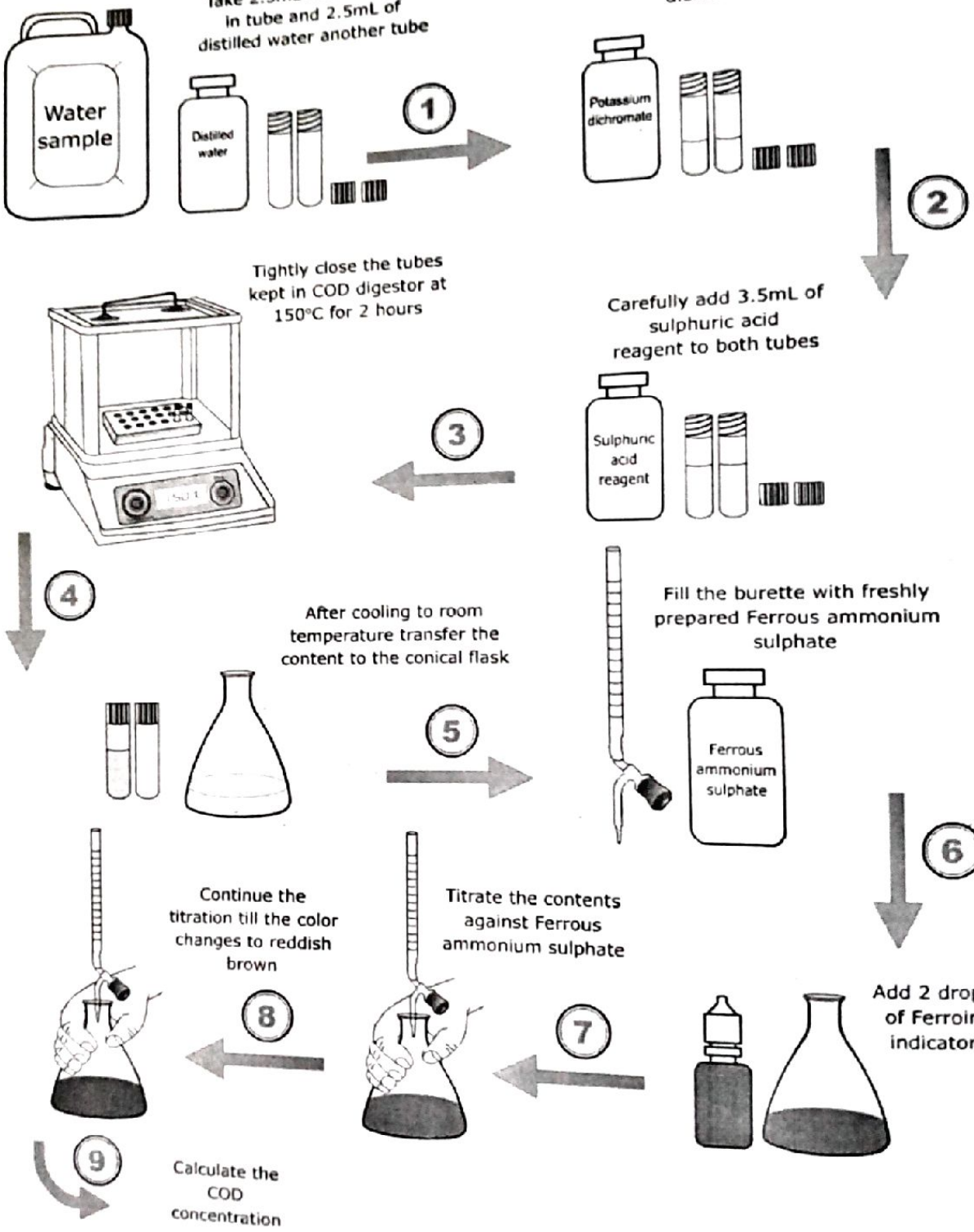
The following precautions should be observed while performing the experiment:

• Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chlorides so that it does not interfere in the determination.

• Nitrites also interfere in the determination of COD and hence during the determination of samples with high concentration of nitrites, 120mg of sulphuric acid is added to the potassium dichromate solution.

• Traces of organic material either from the glassware or atmosphere may cause a positive error. Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.

PROCEDURE CHART



PROCEDURE

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENTS

a) Standard Potassium Dichromate Reagent - Digestion Solution Weigh accurately 4.913 g of potassium dichromate, previously dried at 103ºC for 2 - 4 hours and transfer it to a beaker.

Weigh exactly 33.3g of mercuric sulphate and add to the same beaker.

Measure accurately 167 mL of concentrated sulphuric acid using clean dry measuring cylinder and transfer it to the beaker. Dissolve the contents and cool to room temperature. (If not dissolved keep it over night).

Take 1000 mL standard measuring flask and place a funnel over it.

Carefully transfer the contents to the 1000 mL standard flask and make up to 1000 mL using distilled water.

This is the standard potassium dichromate solution to be used for digestion.

b) Sulphuric Acid Reagent - Catalyst Solution

Weigh accurately 5.5 g silver sulphate crystals to a dry clean 1000 mL beaker. To this carefully add about 500 mL of concentrated sulphuric acid and allow to stand for 24 hours (so that the silver sulphate crystals dissolve completely).

c) Standard Ferrous Ammonium Sulphate solution

Weigh accurately 39.2g of ferrous ammonium sulphate crystals and dissolve it in distilled water.

Take 1000 mL standard measuring flask and place a funnel over it.

Carefully transfer the contents to the 1000 mL standard flask and make up to 1000 mL mark using distilled water.

TESTING OF SAMPLE

- Take three COD vials with stopper (two for the sample and one for the blank).
- Add 2.5 mL of the sample to each of the two COD vials and the remaining COD vial is for blank; to this COD vial add distilled water.
- Add 1.5 mL of potassium dichromate reagent - digestion solution to each of the three COD vials.
- Add 3.5 mL of sulphuric acid reagent - catalyst solution in the same manner.
- * **CAUTION:** COD vials are hot now.
- Cap tubes tightly. Switch on the COD Digester and fix the temperature at 150º C and set the time at 2 hours.
- Place the COD vials into a block digester at 150°C and heat for two hours.
- The digester automatically switches off. Then remove the vials and allow it to cool to the room temperature.
- Meanwhile, get ready with the burette for the titration.
- Fill the burette with the ferrous ammonium sulphate solution, adjust to zero and fix the burette to the stand.
- Transfer the contents of the blank vial to conical flask.
- Add few drops of ferroin indicator. The solution becomes bluish green in colour.
- Titrate it with the ferrous ammonium sulphate taken in the burette.

• End point of the titration is the appearance of the reddish brown colour.
• Note down the volume of ferrous ammonium sulphate solution added for the blank (A) is .....**mL.**
• Transfer the contents of the sample vial to conical flask.
• Add few drops of ferroin indicator. The solution becomes green in colour.
• Titrate it with the ferrous ammonium sulphate taken in the burette.
• End point of the titration is the appearance of the reddish brown colour.
• Note down the volume of ferrous ammonium sulphate solution added for the sample (B) is .....**mL.**

12.7 CALCULATION

For determining the Chemical Oxygen Demand in the given water sample, the readings should be tabulated.

12.7.1 TABLE

Sl No.	**Sample**	**Volume of Sample (mL)**	**Burette Reading (mL)** **Initial** **Final**	**Volume of 0.1 N FAS (mL)**
1	BLANK			
2	SAMPLE 1			
3	SAMPLE 2			

Calculation:

Volume of Ferrous Ammonium sulphate for blank (A) =

Volume of Ferrous Ammonium sulphate for Sample (B) =

Normality of Ferrous Ammonium sulphate N = 0.1 N

Volume of Sample V =

Chemical Oxygen Demand = (A - B * N * 8 * 1000) /Volume of sample taken

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert

the sample size from mL to L.

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

DATA SHEET

- ✓ Date Tested :
- ✓ Tested By :
- ✓ Sample Number : 1

  Sample Location :

  Description : Canal / River water/Ground water

RESULT:-

Chemical Oxygen Demand for the given sample 1 (Canal / River water/Ground water) =

CONCLUSION:-

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

EXPERIMENT NO-14

DETERMINATION OF SOLIDS (SUSPENDED, DISSOLVED, SETTLEABLE)

AIM:

To determination of suspended, dissolved and settleable solids.

APPARATUSANDEQUIPMENT:

a. Electrically heated temperature controlled oven
b. Analytical balance
c. Steam bath
d. Evaporating dish-Porcelain (200ml)
e. Pipettes
f. Desiccator
g. Measuring cylinder (100ml)

INTRODUCTION:

All matter except the water contained in liquid materials classified as "solid waste". The usual definition of solids however refers to the matter that remains as residue upon evaporation and drying at 103°C to 105°C.

PRINCIPLE:

Concentration of solids in water depends upon the source of water and the soil strata from which the water is percolating and starts dissolving the soluble salts. Total solid is the term applied to the residue left in the vessel after evaporation of the sample and its drying in the oven at a definite temperature about 103°C to 105°C.

The total solid enclose total suspended solids, is that portion of total solid remain on the filter paper and the total dissolved solid, is that portion which passes through the filter. Dissolved solids are the portion of solids that passes through normal general size of 2mμ under specified conditions and bigger than that will be retained as suspended solids on the filter paper.

Determination of Total solids and dissolved solids

1. Take an evaporating dish and clean it properly to remove all the impurities.
2. Dry it to 103°C in an oven for 1hr and weigh (W1). Weighing should be carried out after transferring the evaporating dish in the desiccator.
3. Take 50 ml of water sample and transfer it in an evaporating dish.
4. Put it on a steam bath and allow the sample to evaporate
5. After complete evaporating, dry the evaporating dish with residue in oven a 103°C for 1 hr
6. Cool the evaporating dish in desiccator and take weight (W2)
7. Take another 50 ml sample and filter it on filter paper to remove suspend solids.
8. Collect the filtrate in evaporating dish
9. Put it on a steam bath and allow the sample to evaporate
10. Dry the evaporating dish in oven at 180°C for 1 hr.
11. Cool the evaporating dish in desiccator and take weight (W4)

OBSERVATION:

1. Weight of empty evaporating dish (dried at 103°C) (W1) =
2. Weight of evaporating dish + residue of sample dried at 103°C (W2) =
3. Weight of empty evaporating dish (dried at 103°C) (W3) =
4. Weight of evaporating dish + residue of filtered sample dried at 180°C (W4) =

Calculation:

Total solids, mg/l A = (W2 – W1) X1000/ ml of sample

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Dissolved solids, mg/l B = (W4 – W3) X1000/ ml of sample

Suspended Solids, mg/l C = $A - B$

Result:

Conclusion:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

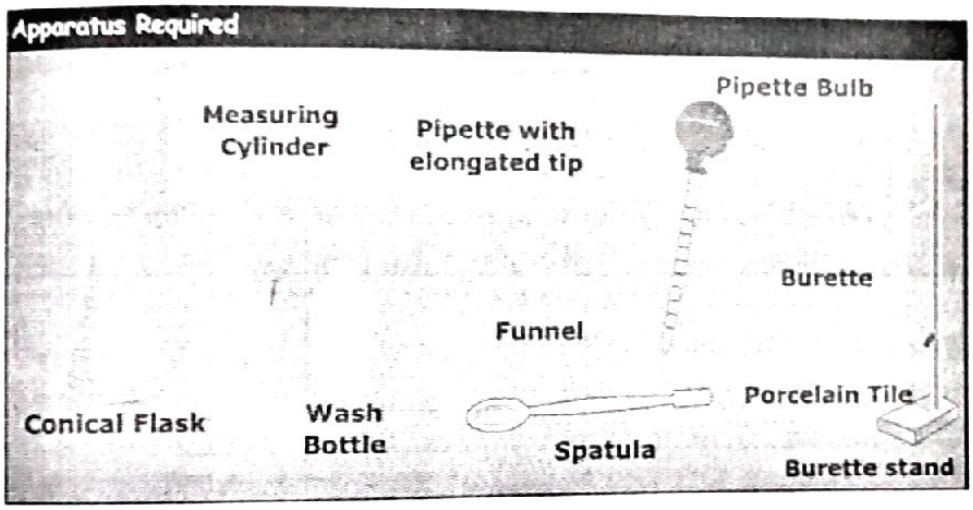
EXPERIMENT NO – 15
A) DETERMINATION OF RESIDUAL CHLORINE

AIM

To determine residual chlorine in the given water sample.

APPARATUS REQUIRED

1. Burette & Burette Stand
2. Porcelain Tile
3. Pipettes with elongated tips
4. Pipette Bulb
5. Wash Bottle
6. 250 mL Graduated Cylinder
7. 500 mL Conical Flask (Erlenmeyer flask)



CHEMICALS REQUIRED

1. Acetic Acid, Conc. (glacial)
2. Potassium Iodide, KI, crystals
3. Sodium thiosulphate
4. Starch indicator
5. Distilled or Deionized Water

PRINCIPLE

The starch-iodide titration method, one of the oldest methods for determining chlorine, is very non-specific for oxidants and generally is used for total chlorine testing at levels above 1 mg/L Cl2.

Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodine is titrated with a standard solution of sodium thiosulphate ($Na_2S_2O_3$) with starch as the indicator.

This method is based on reaction with thiosulfate solution

The end point of the titration is indicated by the disappearance of the blue colored starch-

iodide complex.

ENVIRONMENTAL SIGNIFICANCE

Chlorine residuals determination is used to control chlorination of domestic and industrial wastewaters.

Active chlorine (free and combined) should be determined at each stage in the treatment process of drinking water and in the water mains in order to guarantee bacteriologically impeccable water.

Chlorine determination is important to avoid bad odour and change in the taste of water.

It is determined in the swimming pools to avoid ill effects due to excess chlorination.

Determination of chlorine residual in water distribution is useful to find the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Thus, the main purpose for the chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing micro-organisms.

SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If Analysis is to be carried out with in two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample 0
collection to reduce the change in sample, keep all samples at 4 C.

Do not allow samples to freeze. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- This experiment is a basic experiment and hence there will not be any major difficulties in performing the experiment. The entire procedure should be done in quick time without exposing the solutions to the ambient air.

- Do not expose the potassium iodide crystals in the air. If possible do the experiment in iodine flask instead of conical flask.

- Chlorine in water solutions is not stable. As a result, its concentration in samples decreases rapidly.
- Samples to be analyzed for chlorine cannot be stored or preserved. Tests must be started immediately after sampling. Therefore, samples taken for the chlorine residual test must be grab samples only and excessive agitation must be avoided.
- Exposure to sunlight or other strong light, air, or agitation will further reduce the quantity of chlorine present in solutions.

PROCEDURE

PREPARATION OF REAGENTS

Sodium Thiosulphate solution (0.01N)

Weigh approximately 2.482 g of sodium thiosulphate ($Na_2S_2O_35H_2O$).Transfer to the beaker and dissolve it in boiled distilled water. Transfer it to the standard flask and make it up to 1000 mL.

TESTING OF SAMPLE

- Rinse the burette with sodium thiosulphate and then fill the burette with sodium thiosulphate.
- Fix the burette to the stand.
- Take 200 mL of a given sample in a conical flask.
- Add 5 mL Acetic acid. To acidify the sample. It is used to reduce the pH between 3 and 4 in the conical flask.
- Add about 1 g Potassium Iodide (KI) measured using the spatula and dissolve it by thoroughly mixing it with stirring rod.
- Perform the titration quickly, since iodine liberate faster.
- Titrate the solution with standard $Na_2S_2O_3$ solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 mL of starch solution and continue the titration until the blue color disappears.
- In many cases residual chlorine is very low and starch needs to be added before starting up the titration.
- Note down the burette reading (to know the volume of sodium thiosulphate added).

TABLE

Sample No	Temperature of sample ^0C	Volume of Sample(mL)	Burette Reading (mL) Initial Final	Volume of Titrant (mL) ($Na_2S_2O_3$ solution used)	Residual Chlorine (mg/l)
1. 2. 3.					

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

Calculation:

Volume of Sodium thiosulphate =
Normality of Sodium thiosulphate =
Volume of Sample =
Equivalent weight of Chlorine = 35.45

Residual Chlorine = Volume of Sodium thiosulphate * N * 35.45 * 1000/ Volume of sample taken

*To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Residual Chlorine (mg/L) =

DATA SHEET

- ✓ Date Tested:
- ✓ Tested By :
- ✓ Sample Number : 1
- ✓ Sample Location :
- ✓ Sample Description :
- ✓ Sample Number : 2
- ✓ Sample Location :
- ✓ Sample Description :

RESULTS:

- ✓ The amount of Residual Chlorine in the given sample 1 of water =
- ✓ The amount of Residual Chlorine in the given sample 2 of water =

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

B) DETERMINATION OF AVAILABLE CHLORINE IN BLEACHING POWDER

AIM: To determine the available chlorine in the given sample of the bleaching powder

APPARATUS:

1. Conical flask 2. Burette and pipette

REAGENTS:

1. concentrated acetic acid 2. Potassium iodide crystal
3. Sodium thiosulfate 0.1N 4. Starch indicator

THEORY:

Chlorine is a strong oxidizing agent and liberates iodine from iodide ion.

$$Cl_2 + 2KI \longrightarrow I_2 + 2\,KCl$$

Starch gives blue colour with iodine

The liberated iodine is titrated with standard sodium thiosulfate – a reduing agent.

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2\,NaI$$

The disappearance of blue colour indicates the completion of reaction with free iodine which is converted back to iodine by $Na_2S_2O_3$

PROCEDURE:

1. Take 5 gm (accurately weighed) of fresh bleaching powder adding quantity of water to it, and prepare fine paste. Add some more water stir and allow to settle for a few minutes. Dilute it with distilled water to make up to 1 litre and stopper, the container.
2. Take a known volume (V) of about 25ml of the bleaching powder solution from above in a conical flask and add a pinch of KI.
3. Add 100ml distilled water and 10ml of acetic acid and allow the reaction to complete
4. Titrate the free – iodine liberates and sample with standard 0.1N sodium thiosulfate solution until the yellow colour of the liberated iodine is almost faded out
5. Add 1ml of starch solution and titrate until the blue colour disappears
6. Note down the quantity of sodium thiosulfate added (V1)
7. Repeat the same procedure for distilled water.
8. Note down the volume of the sodium thiosulfate added (V2). V2=0 if distilled water is free from chlorine.

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

ENVIRONMENTAL SIGNIFICANCE:

1. Chlorine is available in different states, gaseous, liquid and also as a solid. Bleaching powder ($CaOCl_2$) is a slaked lime through which chlorine is injected. It is hygroscopic and contains calcium, oxygen and chlorine.

$CaOCl_2 + H_2O \; Ca(OH)_2 + Cl_2$

Bleaching powder loses its chlorine content if exposed to the atmosphere and due to prolonged storage. Hence the amount of chlorine contained by it need be decided before application of bleaching powder to water. Chlorination through bleaching powder is called hypo chlorination which, in India, is almost synonymous to disinfection.

2. This test is useful to asses the quality of bleaching powder. It is also useful to estimate the exact amount of bleaching powder required for effective disinfection of water. If excess chlorine is used, it may irritate eye and nose, bleaching hair, causes allergy and cancers. If the dosage is smaller, pathogens that cause several water borne diseases cannot be killed completely.

CALCULATIONS:

Available chlorine mg/l = (A-B)*N*35.45*1000/ ml of sample taken

(A and B are the ml of thiosulfate required by sample and blank respectively)

Model calculation: if 10ml 0.1N $Na_2S_2O_3$ is used to titrate 25ml of sample, available chlorine =3545 * 10/25 = 1418 mg/l i.e 1418 mg chlorine is present in 5 gm bleaching powder.

Therefore chlorine available = 1.418 g/5 gm. i.e, 28.36 g/100 g i.e, 28.36 %

TABLE:

BLANK READING B=

Identification of sample	Volume of sample taken in ml	Burette reading		Volume of $Na_2S_2O_3$ in ml	Available chlorine
		Initial	final		mg/l %

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.

RESULTS:

CONCLUSION:

Head
School of Civil Engineering
Rajeev Gandhi Memorial College of
Engg. & Tech. (Autonomous)
NANDYAL-518 501.