# Rajiv Gandhi University of Knowledge Technologies 



# Department of Chemical Engineering ENVIRONMENTAL ENGINEERING <br> LABORATORY <br> MANUAL <br> (CH 3702) 

## Course Objectives:

- This lab will provide practical knowledge on characterization of effluents.
- Learn basic water quality parameters.
- To introduce the sampling and monitoring methods.
- To inform the adverse effects of emissions from chemical industries and guidelines set by the environmental protection agencies.


## List of Experiments:

| S.No | Name of the experiment |
| :--- | :--- |
| 1. | Estimation of Ph of the given sample |
| 2. | Determination of Electrical Conductivity of the sample |
| 3. | Determination of Total solids of the given sample |
| 4. | Estimation of hardness of the given sample |
| 5. | Estimation of alkalinity of the given sample |
| 6. | Estimation of Dissolved Oxygen of the given sample |
| 7. | Estimation of Biochemical Oxygen Demand of the given sample |
| 8. | Estimation of Chemical Oxygen Demand of the given sample |
| 9 | Determination of Optimum Coagulant Dosage of the given sample |

## Course Outcomes:

By the end of this course, the student should be able to:

- Understanding fundamentals of water quality.
- Understanding the sources of water pollutants.
- Application of design principles for clarifier devices.
- Understanding the latest techniques for the treatment of effluents and the equipment used.
- Understanding the guidelines set by the environmental protection agencies.


## Experiment 1

Estimation of $\mathbf{p H}$ of the Sample
pH is positive Hydrogen ion concentration. The pH scale is used to express the concentration of hydrogen ions in a liquid. The pH scale ranges from 1 to 14 i.e. most acid to most alkaline. The hydrogen ion concentration is an important parameter in determining the quality of water and wastewater. The pH of the water/wastewater sample is measured by pH meter. A pH meter consists of a measuring probe connected to electronic meter that measures and displays the pH reading.

Principle: The electro chemical potential between a known liquid inside glass electrode and unknown liquid outside. The meaning of pH is potential of hydrogen $(\mathrm{H})$.

Apparatus: pH meter, small beakers

## Procedure:

1. Rinse the probe with distilled water. Calibrate the pH meter using reference solution.
2. Rinse the probe with sample and dip the pH measuring probe in sample and read the value of pH on the screen of the pH meter.
3. Rinse the probe with distilled/deionised water between samples.
4. Thoroughly rinse the probe in distilled water after measurement, keep it in distilled water when not in use.

## Result

Ph of the given sample is :

## Experiment 2 <br> Determination of Electrical Conductivity of the sample

The electrical conductivity (EC) is a measure of the capacity of a substance or solution to carry an electrical current. The conductivity is represented by reciprocal value of electrical resistance in ohms relative to cubic centimeter of water at $25^{\circ} \mathrm{C}$. The measured EC value is used as an alternate method to estimate the total dissolved solids (TDS) concentration of the sample. EC is represented by the symbol ' $k$ ' and its unit are millisiemens per meter ( $\mathrm{mS} / \mathrm{m}$ ) or micromhos per centimeter [ $\mu \mathrm{mho} / \mathrm{cm}$ ].

Principle: In order to measure conductivity, we actually measure resistance of the water sample using a Wheatstone bridge apparatus. Conductance is the inverse of resistance. Conductivity of the sample is obtained by multiplying conductance by the cell constant, which is an unique property of that particular conductivity electrode.

Apparatus: Conductivity meter, small beakers

## Reagent:

Distilled water or deionised water: The water is to have an electrical conductivity of less than $0.01 \mathrm{mS} / \mathrm{m}(<0.1 \mu \mathrm{mho} / \mathrm{cm})$. Boil the water shortly before use to minimize CO2 content (equal to atmospheric equilibrium).
0.01 M Standard potassium chloride solution(KCI): Dissolve 745.6 mg anhydrous KCl (dried 1 hour at $180^{\circ} \mathrm{C}$ ) in conductivity water and dilute to 1000 mL . This solution has an electrical conductivity of $1412 \mu \mathrm{mhos} / \mathrm{cm}$ at $25^{\circ} \mathrm{C}$.

## Procedure:

1. Rinse conductivity cell with 0.01 M KCl solution. Calibrate the conductivity meter using the KCl reference solution to obtain cell constant.
2. Measure the electrical conductance of the 0.01 M KCl solution at room temperature.
3. Calculate the cell constant.
4. Rinse cell with sample. Measure the electrical conductance of the sample.
5. Thoroughly rinse the cell in distilled water after measurement, keep it in distilled water when not in use.

## Calculations

## 1. Compute the cell constant, C

The conductance ( G in $\mu \mathrm{mho}$ ) of standard potassium chloride $(\mathrm{KCl})$ solution is measured using conductivity meter.

Cell constant ' C ' is calculated by the expression:

$$
\mathrm{C}=(\mathrm{k}) / \mathrm{G} .
$$

Where k : Conductivity
G:Conductance
For 0.01 N KCl solution the ' k ' value is $1412 \mu \mathrm{mhos} / \mathrm{cm}$

## 2.Calculate Conductivity of sample

$\mathrm{k}=\mathrm{Gs} \times \mathrm{C}(\mu \mathrm{mho} / \mathrm{cm})$
Where Gs= measured conductance of the given sample.

## Result

The conductivity of the given sample is....

## Experiment 3 Determination of Total solids of the given sample

Solids refers to residue remaining after a water/waste water sample has been evaporated and dried at a specified temperature $\left(103-105^{\circ} \mathrm{c}\right)$. High concentration of total solids will make drinking water unpalatable and might have an adverse effect on human beings. The main source of solids in water includes industrial discharge, sewage treatment plant, fertilizers, road runoff, soil erosion etc.

Aim: To determine total solids in the water sample.
Principle: Water evaporates at $100^{\circ} \mathrm{C}$ where the solids do not. Apparatus:
beaker or crucible, drying oven, Dessicator, weighing balance. Procedure:

1. Take 50 mL aliquot of the water sample in a pre-weighed beaker
2. Put the sample in an oven at $100-105^{\circ} \mathrm{C}$ overnight, or until the water evaporates
3. Take the beaker out of the oven, and cool it in a desiccator
4. Weigh the beaker
5. From the difference in the weight, the concentration of Total Solids (TS) in the sample may be calculated in $\mathrm{mg} / \mathrm{L}$

## Observation and Calculation

Empty weight of the beaker:
Sample added to the beaker:
Weight of the beaker after drying and desiccation:
TS concentration of the sample in $\mathrm{mg} / \mathrm{L}$ :

## Result

Total solids concentration of sample is....

# Experiment -4 <br> Estimation of hardness of the Water 

Hardness is caused due the presence of multivalent cations, mainly $\mathrm{Ca}^{++}$and $\mathrm{Mg}^{++}$in water. Hard waters have many disadvantages, primarily scale formation (i.e., $\mathrm{CaCO}_{3}$ deposition) and enhanced capacity to precipitate soap. Thus measurement of water hardness is very necessary.Total hardness of water is the sum of $\mathrm{Ca}^{++}$and $\mathrm{Mg}^{++}$ concentration in water. The results are expressed as calcium carbonate, in $\mathrm{mg} / \mathrm{L}$, i.e., " $\mathrm{mg} / \mathrm{L}$ as $\mathrm{CaCO}_{3}$ ".

Aim: To determine total hardness of the sample

Principle: Total hardness may be determined by performing a complexometric titration with EDTA as the chelating agent.The indicator Eriochrome Black-T (EBT) is normally blue in color, but becomes red in color when it is complexed with calcium or magnesium. Thus when EBT is added to a solution containing hardness, it complexes $\mathrm{Ca}^{++}$and/or $\mathrm{Mg}^{++}$ and becomes red in color.

When EDTA, which has much stronger affinity for $\mathrm{Ca}^{++}$and $\mathrm{Mg}^{++}$than EBT, is added to the solution, it chelates the $\mathrm{Ca}^{++}$and $\mathrm{Mg}^{++}$ions complexed with EBT. When all such ions are chelated, i.e., the endpoint of the titration is reached; EBT reverts to its original blue color.
Relevant equation: EDTA $+\mathbf{C a}^{++} \boldsymbol{\rightarrow} \mathbf{C a}$ - EDTA

Apparatus: Burette, pipettes, conical flasks, beakers, measuring cylinders

## Reagents

Buffer solution for hardness determination: Dissolve 1.179 g of EDTA disodium salt of EDTA dihydrate and $780 \mathrm{mg} \mathrm{MgSO} 4.7 \mathrm{H}_{2} \mathrm{O}$ or $644 \mathrm{mg} \mathrm{MgCl} 2.6 \mathrm{H}_{2} \mathrm{O}$ in 50 mL of distilled water. Add this solution to $16.9 \mathrm{~g} \mathrm{NH}_{4} \mathrm{Cl}$ and 143 mL conc. $\mathrm{NH}_{4} \mathrm{OH}$ with mixing and dilute to 250 mL with distilled water. Store in a plastic bottle

EDTA titrant for hardness determination, 0.01 M : Dissolve 3.723 g analytical reagent grade EDTA disodium salt dihydrate in distilled water and dilute to 1000 mL .

Indicator: Eriochrome black T

## Procedure

1. Take 50 ml water sample in a conical flask.
2. Add 2 mL of the buffer solution and a pinch of Eriochrome Black-T powder (indicator). Red color complex will be formed.
3. Titrate it using the standard EDTA solution (in burette).
4. At the end point the aliquots change color from red to blue.
5. Note down the volume of EDTA required.
6. Repeat the titration and take the average value.

Observations and calculations:

| S. | Volume of water sample (ml) | Burette Readings |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial(x) | Final(y) |  |
|  | 50 ml |  |  |  |
|  | 50 ml |  |  |  |

1. Determination of molarity of $\mathbf{C a}^{+2}$ and $\mathbf{M g}^{+2}$ sample solution:

$$
\begin{aligned}
& \text { EDTA solution } \\
& \text { Sample solution } \\
& \text { M1 }=0.01 \\
& \mathrm{M} 2=\text { ? } \\
& \mathrm{V} 1=\mathrm{x}-\mathrm{y} \\
& \mathrm{M} 1 \mathrm{~V} 1=\mathrm{M} 2 \mathrm{~V} 2 \\
& \text { M2=? }
\end{aligned}
$$

2. Estimation of total hardness of water or Estimation of $\mathbf{C a}^{+2}$ and $\mathbf{M g}^{+2}$ present in the water sample:

Weight of $\mathrm{Ca}^{+2}$ and $\mathrm{Mg}^{+2}$ per $1000 \mathrm{ml}(1 \mathrm{lit})$
$=\mathrm{M} 2 \times\left(\mathrm{M} . \mathrm{wt}\right.$ of $\mathrm{Ca}^{+2}$ and $\mathrm{Mg}^{+2}$ )
$=(\mathrm{M} 2 \times 100) \mathrm{g}$ of CaCO 3 equivalent
$=\ldots . . . . . \mathrm{mg} /$ litre (or) ppm

## Result=

Weight of $\mathrm{Ca}^{+2}$ and $\mathrm{Mg}^{+2}$ present in 1000 ml of given solution $\quad=\ldots . \mathrm{mg} / \mathrm{l}$ as CaCO 3 ie. Total hardness of the given water sample as $\mathrm{CaCO}_{3}$ is $=\ldots . . \mathrm{ppm}$

## Experiment 5 Estimation of Alkalinity of the given Sample


#### Abstract

Alkalinity is defined as the acid neutralizing capacity of a water sample. Determination of acid neutralizing capacity or alkalinity is important, because of our concern regarding the change in pH (i.e., acidification) of water samples due to acid addition (e.g., acid rain). A natural water sample with high alkalinity will be able to neutralize large amounts of acid without acidification (i.e., lowering of pH ).

Alkalinity of natural waters is mostly due to the presence of hydroxyl $\left(\mathrm{OH}^{-}\right)$, carbonate $\left(\mathrm{CO}_{3}{ }^{2-}\right)$ and bicarbonate ions $\left(\mathrm{HCO}_{3}{ }^{-}\right)$. Alkalinity due to the presence of $\mathrm{OH}^{-}$ions is known as caustic alkalinity, alkalinity due to the presence of $\mathrm{HCO}_{3}{ }^{-}$ions is known as bicarbonate alkalinity, alkalinity due to the presence of $\mathrm{CO}_{3}{ }^{2}$ ions is known as carbonate alkalinity.


## Aim:

To determine the amount of alkalinity in the supplied sample of water

## Principle:

Alkalinity can be obtained by neutralizing $\mathrm{OH}-, \mathrm{CO} 32-$ and $\mathrm{HCO} 3-$ with standard H 2 SO 4 . Titration to pH 8.3 or decolourisation of phenolphthalein indicator will show complete neutralization of $\mathrm{OH}-$ and $1 / 2 \mathrm{CO} 32-$, while to pH 4.4 or sharp change from yellow to pink of methyl orange indicator will indicate total alkalinity.

$$
\begin{gathered}
\mathrm{Ca}(\mathrm{OH}) 2+\mathrm{H} 2 \mathrm{SO} 4 \rightarrow \mathrm{CaSO} 4+2 \mathrm{H} 2 \mathrm{O} 2 \mathrm{CaCO} 3+\mathrm{H} 2 \mathrm{SO} 4 \rightarrow \\
\mathrm{Ca}(\mathrm{HCO} 3) 2+\mathrm{CaSO} 4 \mathrm{Ca}(\mathrm{HCO} 3) 2+\mathrm{H} 2 \mathrm{SO} 4 \rightarrow \mathrm{CaSO} 4+\mathrm{CO} 2
\end{gathered}
$$

## Apparatus required:

Beakers, pipettes, volumetric flasks ,conical flask

## Chemicals required:

0.02 N sulphuric acid, phenolphthalein indicator, methyl orange indicator

## Preparation of reagents used for the present experiment:

## a. 0.02 N H2SO4:

Prepare 0.1 N H2SO4 by diluting 3 ml of con. H2SO4 to 1000 ml . It is standardized against standard 0.1 N Na 2 CO 3 by methyl orange indicator. Now the volume of H 2 SO 4 is diluted to 1000 ml to obtain standard 0.02 N H2SO4.
b. Phenolphthalein indicator:
0.5 g of phenolphthalein is dissolved in 500 ml of $95 \% \mathrm{C} 2 \mathrm{H} 5 \mathrm{OH}$. Add 500 ml of distilled water. 0.02 N NaOH is added dropwise till faint pink colour appears.
c. Methyl orange indicator:
0.5 g of it is dissolved in 1000 ml of water with CO 2 free distilled water.

## Procedure:

1. 50 ml of sample $(\mathrm{H} 2 \mathrm{O})$ is taken in a conical flask and to this $2-3$ drops of phenolphthalein indicator is added
2. Pink colour develops and, it is titrated against 0.02 N H2SO4 till it disappears. Volume of H 2 SO 4 required (A) is noted.
3. To the same flask $2-3$ drops of methyl orange indicator is added and the titration is continued till the orange colour changes to pink. The volume of H 2 SO 4 required
(B) is noted.
4. The experiment is repeated to get concurrent readings.

## OBSERVATIONS AND CALCULATIONS

## Titration 1 (For Phenolphthalein alkalinity)

Burette solution: $\mathrm{H}_{2} \mathrm{SO}_{4}$
Pipette solution: Sample
Indicator: phenolphthalein
End Point: Disappearance of pink colour

| S.No | Volume of water sample$(\mathrm{ml})$ | Burette Readings |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial(x) | Final(y) |  |
| 1. | 50 ml |  |  |  |
| 2. | 50 ml |  |  |  |

Titration 2 (For total alkalinity)
Burette solution: $\mathrm{H}_{2} \mathrm{SO}_{4}$
Pipette solution: Sample
Indicator: methyl orange
End Point: Appearance of pink colour

| S.No | Volume of water sample(ml) | Burette Readings |  | Volume of H2SO4 added (x$-\mathrm{y})$V2 (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial(x) | Final(y) |  |
| 1. | 50 ml |  |  |  |
| 2. | 50 ml |  |  |  |

Phenolphthalein alkalinity as CaCO 3 in $\mathrm{mg} / \mathrm{l}=(\mathrm{V} 1 * \mathrm{~N}$ of $\mathrm{H} 2 \mathrm{SO} 4 * 50,000) / \mathrm{Vol}$. of sample.
Total alkalinity as CaCO 3 in $\mathrm{mg} / \mathrm{l}=(\mathrm{V} 2 * \mathrm{~N}$ of $\mathrm{H} 2 \mathrm{SO} 4 * 50,000) / \mathrm{Vol}$. of sample

## Result

Total alkalinity present in the water sample $=$

## Experiment 6 Estimation of Dissolved Oxygen of the given sample

Dissolved oxygen is one of the most important constituents of natural water system. It indicates the pollution status of river. The dissolved oxygen in water depends upon its temperature, solubility. If DO is less, then it indicates the presence of organic matter. At least 4 $\mathrm{mg} / \mathrm{L}$ of DO is required for fish and other species.

Aim: To determine the amount of dissolved oxygen present in the given sample
Principle: It is based on the principle that oxygen present in the sample oxidizes the divalent manganese to its higher valency under alkaline conditions and that manganese in higher states of valencies is capable of oxidizing $\mathrm{I}^{-}$to $\mathrm{I}_{2}$ under acidic conditions. Thus the amount of $\mathrm{I}_{2}$ released is equivalent to the dissolved oxygen present. The iodine is measured with standard sodium thiosulphate solution.

If no oxygen is present, a pure white precipitate of $\mathrm{Mn}(\mathrm{OH})_{2}$ forms

$$
\mathrm{Mn}_{2}+2 \mathrm{OH}^{-} \rightarrow \mathrm{Mn}(\mathrm{OH})_{2}(\mathrm{~s})(\text { white precipitate })
$$

If oxygen is present

$$
\mathrm{Mn}_{2}^{+}+2 \mathrm{OH}^{-}+\frac{1}{2} \mathrm{O}_{2} \rightarrow \mathrm{MnO}_{2}(\mathrm{~s})+\mathrm{H}_{2} \mathrm{O} \mathrm{MnO}_{2}+
$$

Under low pH

$$
2 \mathrm{I}^{-}+4 \mathrm{H}^{+} \rightarrow \mathrm{Mn}_{2}^{+}+\mathrm{I}_{2}+2 \mathrm{H}_{2} \mathrm{O}
$$

$\mathrm{I}_{2}$ is rather insoluble in water, but forms a complex with the excess iodide present reversibly forms the more soluble tri-iodate thus preventing escape of $\mathrm{I}_{2}$ from the solution $\mathrm{I}_{2}+$
$\mathrm{I}^{-} \leftrightarrow \mathrm{I}_{3}{ }^{-}$
Apparatus: 300 mL BOD bottles, burette, pipette, conical flask etc.,

## Reagents

Manganous sulfate solution : Dissolve $48 \mathrm{~g} \mathrm{MnSO}_{4} 4 \mathrm{H}_{2} \mathrm{O}$, or $40 \mathrm{~g} \mathrm{MnSO}_{4} 2 \mathrm{H}_{2} \mathrm{O}$, or 36.4 g $\mathrm{MnSO}_{4} \mathrm{H}_{2} \mathrm{O}$ in distilled water, filter and dilute to 100 ml . This solution should not produce a blue color with starch indicator when added to an acidified potassium iodide (KI) solution.

Alkaline-iodide-sodium azide solution: Dissolve 50 g NaOH (or 70 g KOH ) and 13.5 g
NaI (or 15 g KI ) in distilled water and dilute to 100 ml . Add $1 \mathrm{~g} \mathrm{NaN}_{3}$ dissolved in 4 mL distilled water. Mix both.

Starch indicator solution: Use either an aqueous solution or soluble starch powder mixture. Prepare an aqueous solution by dissolving 2 g of laboratory grade soluble starch powder and 0.2 g of salicylic acid (as a preservative) in 100 mL of hot distilled water.

Sodium thiosulfate standard solution, $\mathbf{0 . 0 2 5 0} \mathbf{N}$ : Dissolve $6.205 \mathrm{~g} \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3} 5 \mathrm{H}_{2} \mathrm{O}$ in distilled water. Add 1.5 mL 6 N NaOH or 0.4 g solid NaOH and dilute to 1 liter.

## Procedure

1. Collect the sample to be tested in a 300 mL BOD bottle taking special care to avoid adding air to the liquid being collected. Fill bottle completely and add stopper.
2, Remove bottle stopper and add 2 mL of the manganous sulfate solution at the surface of the liquid. Add 2 mL of the alkaline-potassium iodide-sodium azide solution at the surface of the liquid. Replace the stopper, avoid trapping air bubbles and shake well by inverting the bottle several times.
2. Repeat shaking after floc has settled halfway. Allow floc to settle a second time.
3. Add 2 mL of concentrated sulfuric acid and close the bottle with stopper. Rinse the top of the bottle to remove any acid and shake well until the precipitate is completely dissolved (uniform yellow color).
4. Take 200 mL of sample from the BOD bottle into a conical flask and titrate with 0.0250 N sodium thiosulfate until the solution is a pale yellow (straw) color.
5. Add a small quantity (approximately 1 mL ) of starch indicator continue the titration with 0.0250 N sodium-thiosulfate until blue colour disappears (blue to colorless). Record the mL of sodium-thiosulfate used.

## Observations and Calculations

Calculate the concentration of DO in the sample using the following formula:
$D O \mathrm{mg} / \mathrm{L}=\frac{\mathrm{mL} \text { of titrant } * \text { normality of titrant } * 8000}{\text { volume of sample titrated }}$

| Trial | Burette reading mL of $\left(\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3} 5 \mathrm{H}_{2} \mathrm{O}\right)$ |  |  |  |
| :--- | :--- | :--- | :--- | :---: |
|  | Initial reading (IR) | Final reading (FR) | IR-FR |  |
|  |  |  |  |  |
| II |  |  |  |  |
| Average |  |  |  |  |

## Result

DO of the given sample $=$

## Experiment 7 <br> Estimation of Biochemical Oxygen Demand of the given sample

Biochemical Oxygen Demand (BOD): BOD is defined as the amount of oxygen required by bacteria while stabilizing decomposable organic matter under controlled aerobic conditions. This test is widely used to determine the pollution strength of domestic and industrial wastes in terms of the oxygen demand. The BOD test relies on measurable depletion of dissolved oxygen (DO) over a specified period of time, generally 5 day at $20^{\circ} \mathrm{C}$ incubation. The BOD is considered complete after 20 days. 20 Days is too long to wait, 5 days is a reasonable period for most of the BOD to be exerted is about 70 to $80 \%$ of total BOD.

Aim: To determine the BOD of the given sample
Principle: The BOD is measured by determining the oxygen consumed (by the bacteria) from a sample placed in an air-tight container and kept in a controlled environment for a preselected period of time.

Apparatus: BOD bottles, Burette, pipette, conical flask, BOD Incubator

## Reagents

Manganous sulfate solution : Dissolve 480 g MnSO4 4H2O, or 400 g MnSO 42 H 2 O , or 364 g MnSO4 H2O in distilled water, filter and dilute to 1 liter. This solution should not produce a blue color with starch indicator when added to an acidified potassium iodide (KI) solution.

Alkaline-iodide-sodium azide solution: Dissolve 500 g NaOH (or 700 g KOH ) and 135 g NaI (or 150 g KI) in distilled water and dilute to IL. Add 10 g NaN3 dissolved in 40 mL distilledwater.Mix both solution.

Starch indicator solution: Use either an aqueous solution or soluble starch powder mixture. Prepare an aqueous solution by dissolving 2 g of laboratory grade soluble starch powder and
0.2 g of salicylic acid (as a preservative) in 100 mL of hot distilled water.

Sodium thiosulfate standard solution, $\mathbf{0 . 0 2 5 0}$ N: Dissolve $6.205 \mathrm{~g} \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3} 5 \mathrm{H}_{2} \mathrm{O}$ in distilled water. Add 1.5 mL 6 N NaOH or 0.4 g solid NaOH and dilute to 1 liter.

## Procedure

1. Saturate water with oxygen by bubbling air through it by using a compressor. Dissolved oxygen (DO) concentration in this water should be at least $8 \mathrm{mg} / \mathrm{L}$. This is known as dilution water.
2. Prepare a blank sample (using 300 mL of dilution water only) in a BOD bottle. Measure the DO (B1) of the sample (using DO Experiment). Incubate the blank sample for 5 days at $\quad 20^{\circ} \mathrm{C}$. Measure DO after incubation (B2). The DO of the dilution water should not be much different from the initial value.
3. Prepare two samples by adding 10 mL of the wastewater in the BOD bottle, and making up to 300 mL with dilution water. Measure initial DO(D1). Incubate the sample for 5 days at $20^{\circ} \mathrm{C}$ and Measure final DO(D2) in each sample.
4. Calculate BOD5.

## Observations and calculations

I) BLANK

| Trial | Burette reading mL of (Na2S2O3 5H2O) |  |  | DO in mg/L |
| :--- | :--- | :--- | :--- | :--- |
|  | Initial reading (IR) | Final reading (FR) | IR-FR |  |
| $0^{0^{\text {III }} \text { day }}$ |  |  |  |  |
| $5^{\text {UII }}$ day |  |  |  |  |

## ii) SAMPLE

$\mathbf{0}^{\text {th }}$ day reading

| Trial | Burette reading mL of (Na2S2O3 5H2O) | DO in mg/L |  |  |
| :--- | :--- | :--- | :--- | :--- |
|  | Initial reading (IR) | Final reading (FR) | IR-FR |  |
| II |  |  |  |  |
| II |  |  |  |  |
| Average |  |  |  |  |

$\mathbf{5}^{\text {th }}$ day reading

| Trial | Burette reading mL of (Na2S2O3 5H2O) | DO in mg/L |  |  |
| :--- | :--- | :--- | :--- | :--- |
|  | Final reading (FR) | Initial reading (IR) | FR-IR |  |
| II |  |  |  |  |
| II |  |  |  |  |
| Average |  |  |  |  |

## 5 day BOD, $=$ BOD5 $(\mathrm{mg} / \mathrm{l})=\{[\mathrm{D} 1-\mathrm{D} 2]-[\mathrm{B} 1-\mathrm{B} 2]\}^{*} \mathbf{P}$

$\mathrm{D} 1=\mathrm{DO}$ of the sample immediately after preparation, $\mathrm{mg} / \mathrm{L}, \mathrm{D} 2=$ DO of the sample after 5 day incubation at $20^{\circ} \mathrm{C}, \mathrm{mg} / \mathrm{L}, \mathrm{B} 1=\mathrm{DO}$ of blank immediately after preparation, $\mathrm{mg} / \mathrm{L}$,
$\mathrm{B} 2=\mathrm{DO}$ of blank after 5 day incubation at $20^{\circ} \mathrm{C}, \mathrm{mg} / \mathrm{L}, \mathrm{P}=$ dilution factor

## Result

BOD of the given sample

## Experiment 8 Estimation of Chemical Oxygen Demand of the given sample

Chemical oxygen demand (COD) test is used to measure the oxygen equivalent of the organic material in waste water that can be oxidized chemically using dichromate in an acid solution. COD is widely used as a means of measuring the organic strength of domestic and industrial wastes. It allows measurement of a waste in terms of the total
quantity of oxygen required for oxidation to $\mathrm{CO}_{2}$ and $\mathrm{H}_{2} \mathrm{O}$. The major advantage of COD test is the short time required for evaluation. Many organic matter which are difficult to oxidized biologically such as lignin can be oxidized chemically.

Aim: To determine the Chemical oxygen demand (COD) of the given sample.
Principle: It is based on the principle that organic matter present in the sample gets oxidized completely by potassium dichromate $\left(\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}\right)$ in the presence of sulphuric acid $\left(\mathrm{H}_{2} \mathrm{So} \mathrm{O}_{4}\right)$ and catalyst silver sulphate $(\mathrm{AgSo} 4)$ to produce $\mathrm{CO}_{2}$ and $\mathrm{H}_{2} \mathrm{O}$. The excess of potassium dichromate remaining after the reaction is treated with ferrous ammonium sulphate. The dichromate consumed gives $\mathrm{O}_{2}$ required for oxidation of organic matter.

Apparatus: COD digestion vessel, COD digester, burette, pipette and beaker.

## Reagents

Standard potassium dichromate solution:, $0.0417 \mathrm{M}(0.25 \mathrm{~N})$ : Dissolve 12.259 g
$\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$, primary standard grade, previously dried at $103^{\circ} \mathrm{C}$ for 2 hours, in distilled water and dilute to 1 L .

Sulphuric acid reagent: Add $5.5 \mathrm{~g} \mathrm{Ag}_{2} \mathrm{SO}_{4}$ technical or reagent grade, per kg of conc. $\mathrm{H}_{2} \mathrm{SO}_{4}$, keep for a day or two to dissolve.

Ferroin indicator solution: Dissolve 1.485g 1, 10-phenanthroline monohydrate and 695 $\mathrm{mg} \mathrm{FeSO}_{4} .7 \mathrm{H}_{2} \mathrm{O}$ in distilled water and dilute to 100 mL . Commercial preparation may also be available.

Standard ferrous ammonium sulphate (FAS): titrant, 0.25 M : Dissolve 98 g $\mathrm{Fe}\left(\mathrm{NH}_{4}\right)_{2}\left(\mathrm{SO}_{4}\right)_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O}$ in distilled water, add 20 mL conc. $\mathrm{H}_{2} \mathrm{SO}_{4}$, cool and dilute to 1 L .

## Procedure

1. Take 2.5 ml of the sample in digestion vessel and add 1.5 ml of 0.0417 N of
$\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution and 3.5 ml of $\mathrm{Ag}_{2} \mathrm{SO}_{4}+\mathrm{H}_{2} \mathrm{SO}_{4}$ solution.
2. Reflux the mixture on a hot plate/digester for 2 hrs at $150^{\circ} \mathrm{c}$ and cool the mixture to room temperature.
3. Titrate mixture against ferrous ammonium sulphate (FAS) solution using ferroin indicator till blue green color turns to reddish brown color. Note down the volume
of FAS used as 'A'mL.
4. Repeat the experiment for blank sample (distilled water), note down the volume of FAS used as 'B'mL.

## Observations and calculations:

COD $(\mathrm{mg} / \mathrm{L})=[\{(\mathrm{B}-\mathrm{A}) * \mathrm{M} * 8000\}] /$ Volume of Sample
where:
A = FAS used for sample, mL B
= FAS used for blank, mL M =
Molarity of FAS

| sample | Burette reading |  | Volume of FAS <br> consumed(ml) |
| :--- | :--- | :--- | :--- |
|  | Initial | Final |  |
| Blank |  |  |  |
| Sample |  |  |  |
|  |  |  |  |

## Result

The COD of the given water sample ......

## Experiment-9

## Determination of Optimum Coagulant Dosage of the given sample

Coagulation is the process of destabilizing colloidal particles so that particle growth can occur as a result of particle collisions. In wastewater treatment settleable solids are effectively removed by sedimentation process. But, the small size colloidal particles are not possible to remove by gravitational settling due to Brownian motion. However, if the colloidal particles are destabilized through agglomeration of particles into group/large particles, increase in settling velocities, they can be effectively removed by sedimentation tank. The theory of chemical coagulation is very complex. The exact mechanism of coagulation is not known, however, following four mechanisms are thought to be occurring during coagulation process. These include ionic layer compression, adsorption, charge neutralization and inter-particle bridging. Typical coagulants used in wastewater treatment are synthetic organic polymers, metal salts such as alum or ferric sulphate, prehydrolized metal salts (polyaluminum chloride and polyiron chloride).

Aim: To find the optimum amount of coagulant required to treat given water sample.
Principle: Metal salts hydrolyse in presence of the natural alkalinity to form metal hydroxides. The divalent cations can reduce the zeta-potential, while the metal hydroxides are good absorbents and hence remove the suspended particles by enmeshing them.

Alum $\left[\mathrm{Al}_{2} \mathrm{~S}\left(\mathrm{SO}_{4}\right)_{3} .18 \mathrm{H}_{2} \mathrm{O}\right]$ is the most widely used coagulant in water treatment. When alum solution is added to water, the molecules dissociate to yield $\mathrm{SO}_{4}{ }^{2-}$ and $\mathrm{Al}^{3+}$. The +ve species combine with negatively charged colloidal to neutralise part of the charge on the colloidal particle. Thus, agglomeration takes place.

Coagulation is a quite complex phenomenon and the coagulant should be distributed uniformly throughout the solution. A flash mix accomplishes this.

Apparatus: Jar test apparatus, turbid meter, beakers, pipette and pH meter.

## Reagents

Alum, Ferric chloride

## Procedure

1. Measure the initial turbidity of the given sample.
2. Find the pH of the sample and adjust it to 6 to 8.5 .
3. Take six jars of 11 capacity and take 0.51 of sample in each jar and add varying doses of alum $(1,2,4,6,8,10 \mathrm{mg} / \mathrm{l})$ to each one of them.
4. Keep the sample in jar test apparatus and put it for rapid mixing at 100 rpm for 1 min and then to slow mixing at 20 rpm for 20 min .
5. Stop the motor and allow the sample to settle for 30 min . Collect the supernatant from each jar and find out the turbidity.
6. Plot a graph with alum dosage along x -axis and turbidity along y -axis.
7. The dosage of alum, which represents least turbidity, gives Optimum

Coagulant Dosage (O.C.D.).

## Observations and calculations

| Concentration (ppm) | Turbidity (NTU) |
| :--- | :--- |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

## Result

Optimum coagulant dosage $=$ $\mathrm{mg} / \mathrm{L}$

